Atomic Absorption Analysis Cookbook

Section 8

Non-ferrous Metals Analysis

SHIMADZU CORPORATION

KYOTO, JAPAN

Atomic Absorption Analysis Cookbook

Section 8

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Introduction

Cookbook Section 8 describes the methods used for analysis of non-ferrous metals.

These analysis methods, detailed in the Japan Industrial Standards, are for elements to be analyzed by the atomic absorption method. They were modified to provide the optimum results when using a Shimadzu atomic absorption spectrophotometer.

These analysis methods assume that the sample compositions conform to Japan Industrial Standards specifications, but for cases in which the actual sample compositions differ from the Japan Industrial Standards, the pretreatment method, interference at time of measurement, background absorption and flame conditions may not necessarily apply.

The measurement conditions provided here are appropriate when using an AA-6000 Series Atomic Absorption Spectrophotometer. When using another atomic absorption spectrophotometer, the calibration curve concentration range and measurement conditions must be corrected.

15. Non-ferrous Metals Analysis

15.1 Copper and Copper Alloy Analysis Method

Reference materials:

Japan Industrial Standard, Copper and Copper Alloy Atomic Absorption Analysis Method, JIS H 1291

15.1.1 Sample Pretreatment

Weigh out 1.0g of sample to the nearest 1mg, transfer it to a 200m*l* beaker, cover with a watch glass. After adding 20m*l* of an acid mixture containing 1 part hydrochloric acid, 1 part nitric acid, and 2 parts water, heat gently to decompose. Rinse the bottom of the watch glass ,and the inner wall of the beaker with about 10m*l* of water, and heat gently to drive off the nitric acid compounds. After cooling, rinse into a 100m*l* volumetric flask, and dilute to bring up volume.

Note: For silicon pitch copper bronze, weigh out 1.0g of sample into a 100m*l* polyethylene beaker. After decomposing with 20m*l* of the acid mixture, add 5m*l* of hydrofluoric acid (1+9) and mix. Then add 10m*l* of boric acid solution (5 w/v%) and heat for about 15minutes at 70 – 80°C. After cooling, rinse with water into a 10m*l* volumetric flask, and dilute to bring up to volume.

15.1.2 Flame Atomic Absorption Method

a) Target element and quantitation range

Element	Percent Contained (%)
Al	0.1 – 3.0
Be	0.2 – 2.0
Co	0.01 – 1.0
Cr	0.01 – 2.0
Fe	0.01 – 6.0
Mn	0.01 – 3.0
Ni	0.01 – 7.0
Pb	0.01 – 4.5
Si	0.1 – 4.0
Sn	0.02 – 4.0
Zn	0.01 – 1.0

b) Measurement procedures

Measurement is conducted using the following procedure. Refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions, for the lamp current, slit width and flame conditions.

Reagents:

1) Al standard solution (1000μg Al/m*l*)
2) Be standard solution (1000μg Be/m*l*)
3) Co standard solution (1000μg Co/m*l*)
4) Cr standard solution (1000μg Cr/m*l*)
5) Fe standard solution (1000μg Fe/m*l*)
6) Mn standard solution (1000μg Mn/m*l*)
7) Ni standard solution (1000μg Ni/m*l*)
8) Pb standard solution (1000μg Pb/m*l*)
9) Si standard solution (1000μg Si/m*l*)
10) Sn standard solution (1000μg Sn/m*l*)
11) Zn standard solution (1000μg Zn/m*l*)

For 1) – 11) above, refer to Cookbook Section 2, Item 3 Preparing Standard.

Procedure:

1) Depending on the percent of quantitation element contained, use the apportion ratio of the following table to introduce the appropriate aliquot of pretreated solution into 100m*l* volumetric flask, and bring up to volume with hydrochloric acid (1+9).

   At this time, for a blank test, take an appropriate amount of reagent containing no sample, and perform the same pretreatment as that used for the sample. Use this solution to determine the amount of target element contained using a calibration curve generated for the blank test. The obtained value may be used for correction of the value obtained in sample measurement.

Note: To generate the calibration curve for the blank test, add increasing concentrations of the target element to several volumetric flasks containing 20m*l* of the acid mixture, and then dilute with water to bring up to volume.

Apportion Ratio Example

Element	Percent Contained (%)	Apportion Ratio	Concentration Range (μg/100m*l*)
Al	0.1 – 3.0	1/5	0.2 – 6.0
Be	0.1 – 2.0	1/10	0.1 – 0.2
Co	0.01 – 0.2	1/5	0.02 – 0.4
	0.1 – 1.0	1/20	0.05 – 0.5
Cr	0.01 – 0.2	1/5	0.02 – 0.4
Fe	0.01 – 0.2	1	0.1 – 2.0
	0.1 – 2.0	1/10	0.1 – 2.0
	1.0 – 6.0	1/20	0.5 – 3.0
Mn	0.1 – 0.1	1	0.1 – 1.0
	0.1 – 1.0	1/10	0.1 – 1.0
	0.5 – 3.0	1/20	0.25 – 1.5
Ni	0.01 – 0.2	1	0.1 – 2.0
	0.2 – 2.0	1/10	0.2 – 2.0
	1.0 – 7.0	1/20	0.5 – 3.5
Pb	0.01 – 0.2	1	0.1 – 2.0
	0.2 – 2.0	1/10	0.2 – 2.0
	1.0 – 4.5	1/20	0.5 – 2.5
Si	0.1 – 1.0	1	1.0 – 10
	0.5 – 4.0	1/5	1.0 – 8.0
Sn	0.02 – 1.0	1	0.2 – 10
	1.0 – 4.0	1/5	2.0 – 8.0
Zn	0.01 – 0.1	1/5	0.02 – 0.2
	0.1 – 0.5	1/10	0.1 – 0.5
	0.5 – 1.0	1/20	0.5 – 1.0

2) When generating the calibration curve, prepare a standard solution having as similar composition as possible of copper and the other main constituents as in the sample solution, and introduce increasing concentrations of the copper solution with its other constituents into several 100m*l* volumetric flasks. To each, add increments of the standard solution for each element. Then add hydrochloric acid (1+1) so that its concentration is approximately the same as that in the sample solution. Dilute with water to bring up to volume.

Note: The main constituents in the alloy varies with the type of alloy. Normally, it is a good idea to target many elements. For example, brass contains copper and zinc/phosphorous, bronze contains copper and tin/special aluminum, bronze contains copper, aluminum, iron, manganese and nickel/ German silver contains copper, zinc and nickel.

Example of Standard Solution Addition Amount

1) Brass (copper) containing 59.0 – 62.0% lead and iron (Use apportion ratio 1)

Liquid quantity: 100m*l*

Series	Lead and Iron Addition Amount (mg)	Solution Concentration (μg/m*l*)	Percent Contained (%)	Amount of copper added (mg)	Amount of lead added (mg)
1	0	0	0	600	400
2	0.10	1.0	0.01	600	400
3	0.30	3.0	0.03	600	400
4	0.60	6.0	0.06	600	400
5	0.90	9.0	0.09	600	400
6	1.2	12.0	0.12	600	400

2) Example showing iron, nickel and manganese in special aluminum (Use apportion ratio 1/20)

Liquid quantity: 100ml

Series	Iron			Nickel			Manganese			Amount of copper added	Amount of aluminum added
	Addition amount (mg)	Solution Concentration μg/m*l*	Percent Contained (%)	Addition amount (mg)	Solution Concentration μg/m*l*	Percent Contained (%)	Addition amount (mg)	Solution Concentration μg/m*l*	Percent Contained (%)	(mg)	(mg)
1	0	0	0	0	0	0	0	0	0	40	5
2	0.5	5	1.0	3.5	35	7.0	0.20	2.0	0.4	40	5
3	1.0	10	2.0	2.5	25	5.0	0.40	4.0	0.8	40	5
4	1.5	15	3.0	1.5	15	3.0	0.60	6.0	1.2	40	5
5	2.0	20	4.0	1.0	10	2.0	0.80	8.0	1.6	40	5
6	2.5	25	5.0	0.5	5	1.0	0.10	1.0	2.0	40	5
7	3.0	30	6.0	0.25	2.5	0.5	0.12	12.0	2.4	40	5

Measurement:

1) Al

Wavelength : 309.3nm

Calibration curve range : 2 – 60μg /ml

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 2

2) Be

Wavelength : 234.9nm

Calibration curve range : 0.2 – 2μg /m*l*

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 7

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

3) Co

Wavelength : 240.7nm

Calibration curve range : 0.2 – 5μg /ml

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 12

4) Cr

Wavelength : 357.9nm

Calibration curve range : 0.2 – 5μg /ml

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 13

5) Fe

Wavelength : 248.3nm

Calibration curve range : 0.2 – 5μg /m*l*

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 16

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

6) Mn

Wavelength : 279.5nm

Calibration curve range : 0.2 – 5μg /ml

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 22

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

7) Ni

Wavelength : 232.0nm

Calibration curve range : 0.2 – 5μg /ml

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 26

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

8) Pb

Wavelength : 283.3nm

Calibration curve range : 1 – 20μg /ml

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 27

9) Si

Wavelength : 251.6nm

Calibration curve range : 10 – 200μg /ml

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 34

10) Sn-1

Wavelength : 224.6nm

Calibration curve range : 5 – 100μg /m*l*

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 37

Sn-2

Wavelength : 224.6nm

Calibration curve range : 10 – 200μg /m*l*

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 35

Sn-3

Wavelength : 286.3nm

Calibration curve range : 20 – 300μg /m*l*

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 36

11) Zn

Wavelength : 213.9nm

Calibration curve range : 0.05 – 1μg /m*l*

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 44

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

15.2 Analysis Method for Oxygen Free Copper for Electron Tubes

Reference materials:

Japan Industrial Standard Analysis Method for Oxygen Free Copper for Electron Tubes, JIS H 1202

15.2.1 Sample Pretreatment

a) Bi (Tri-n-octylmethylammonium bromide extraction)

Weigh out 5.0g of sample to the nearest 10mg. Transfer to a 200m*l* beaker, cover with a watch glass, and add 30m*l* of nitric acid (1+1) and heat gently to decompose. Rinse the watch glass and the inner wall of the beaker with a small amount of water. Continue to heat to concentrate, and then heat on a water bath to volatilize until evaporated. To this add 2m*l* of nitric acid (1+1) and 10m*l* of water. Heat to dissolve the salts, and add water to bring the volume to about 50m*l*.

To this solution, accurately add 7.5ml of hydrobromic acid (1+8), add water to bring volume to about 80m*l*. Using a pH meter, add sodium hydroxide solution to adjust the pH to 1.5 – 1.7. Then transfer to a 200m*l* separating funnel and add water to bring volume to 150m*l*. Add exactly 10m*l* of extraction solvent, and after shaking vigorously for about 5 minutes, let stand until contents separate into 2 layers. Then remove the aqueous phase. Pass the organic phase through dry filter paper or degreased cotton. Transfer to a 15 – 20m*l* stoppered test tube, and add butyl acetate to bring volume to 10m*l*.

Note: Extraction solvent: Add butyl acetate to 6ml of tri-n-octylmethylammonium chloride to bring to a volume of 40ml. Transfer this solution to a 100ml separating funnel, add 40ml of hydrobromic acid (1+8). After shaking vigorously, remove the aqueous phase and dilute the organic phase with butyl acetate by a factor of 5.

b) Cd, Zn (Tri-n-octylamine extraction)

Weigh out 2.0g of sample to the nearest 10mg, and transfer to a quartz beaker. Cover with a watch glass, add 10m*l* of nitric acid(1+1), and heat gently to decompose. Rinse the watch glass and the inner wall of the beaker with a small amount of water, add 20m*l* of sulfuric acid (1+1), and continue heating until the white fumes of sulfuric acid are generated to remove the nitric acid.

After cooling, add 40m*l* of hydrochloric acid (1+1) to dissolve the salts, transfer to a 200m*l* separating funnel, and add water to bring volume to 100m*l*. Add 5m*l* of extraction solvent, and after shaking vigorously, let stand until contents separate into 2 layers. Then transfer the aqueous phase to a 200m*l* separating funnel.

To this, add 5m*l* of extraction solvent, and after shaking vigorously for about 5 minutes, let stand until contents separate into 2 layers. Then remove the aqueous phase. Pass the organic phase through dry filter paper or degreased cotton, transfer both to a 15 – 25m*l* stoppered test tube, and add 4-methyl-2-pentanone (MIBK) to bring volume to 15m*l*.

Note: Extraction solvent: Dissolve 2m*l* of tri-n-octylamine and dilute to 100m*l*.

c) Pb (Tri-n-octylmethylammonium bromide extraction)

Weigh out 3.0g of sample to the nearest 10mg, transfer to a 200m*l* beaker, cover with a watch glass, add 10m*l* of nitric acid (1+1) and heat gently to decompose. Rinse the watch glass and the inner wall of the beaker with a small amount of water, add 40m*l* of sulfuric acid (1+1), and continue heating until the white fumes of sulfuric acid are generated to completely drive off the nitric acid.

After cooling, add 70m*l* of water, then add exactly 30m*l* of hydrobromic acid (1+8), and gently heat to dissolve the salts. After cooling, transfer to a 200m*l* separating funnel, and add water to bring volume to 150m*l*. Add exactly 10m*l* of extraction solvent, and after shaking vigorously for about 5 minutes, let stand until contents separate into 2 layers. Then remove the aqueous phase. Pass the organic phase through dry filter paper or degreased cotton, transfer to a 15 – 20m*l* stoppered test tube, and add butyl acetate to bring volume to 10m*l*.

Note: Extraction solvent: Same procedure as that for Bi (Tri-n-octyl-methyl-ammonium bromide extraction)

d) Pb (Iron hydroxide precipitation separation)

Weigh out 20g of sample to the nearest 10mg, transfer to a 500m*l* beaker, cover with a watch glass. After adding 160m*l* of nitric acid to decompose, boil gently to drive off the nitrogen oxide. Then add water to bring up to 300m*l*. Add 5m*l* of ferric ammonium sulfate (10 w/v%), and while mixing, add aqueous ammonia to dissolve the copper hydroxide precipitate.

After it dissolves, add 50m*l* more. Next add 15g of ammonium carbonate and heat. After boiling gently for about 5 minutes, set aside in a warm location (60 – 80°C) for 1 – 2 hours. Separate the precipitate using Type 5A filter paper, and after rinseing several times with warm ammonia, rinse solution (made by adding 15g of ammonium carbonate to 500m*l* of ammonia (2+50)), transfer it to the original beaker by rinseing with warm water. Place the beaker under the filter, and drip 10m*l* of hydrochloric acid over the filter paper to dissolve the precipitate on the filter paper and in the beaker. Rinse the filter paper with warm hydrochloric acid (1+50).

Heat the combined filtrate and rinse solution in the beaker to concentrate it. After its volume is decreased to about 10m*l*, transfer it to a 100m*l* beaker using hydrochloric acid (1+50). Add 2m*l* of sulfuric acid (1+1) and heat the concentrate to dryness. After cooling, add 8m*l* of hydrochloric acid (1+1), heat to dissolve. After cooling, transfer to a 25m*l* volumetric flask using hydrochloric acid (1+50), and dilute up to volume.

15.2.2 Flame Atomic Absorption Method

a) Target element and quantitation range

Bi	Tri-n-octylmethylammonium bromide extraction	0.00002%
Cd	Tri-n-octylamine extraction	0.00002%
Pb	Tri-n-octylmethylammonium bromide extraction	0.00005%
	Iron hydroxide precipitation separation	0.0001%
Zn	Tri-n-octylamine extraction	0.00002%

b) Measurement procedures

Measurement is conducted using the following procedure. To generate the calibration curve, refer to Cookbook Section 2, Item 3 Preparing Standards.

- Bi

Reagent:

Bi standard solution (100μg of Bi/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to section a) above can be used for measurement as it is. At this time, take an appropriate amount of reagent containing no sample, perform the same

pretreatment as that used for the sample, and then measure this solution. The obtained value may be used for the correction of the value obtained in sample measurement.

2) For the standard solutions used to generate the calibration curve, take several aliquots of Bi standard solution (100μg of Bi/m*l*) with increasing concentrations of Bi from 0 – 50μg, and to each of these, add exactly 30m*l* of sodium nitrate solution (8.5 w/v%) and 7.5m*l* of hydrobromic acid (1+8). Transfer these solutions to 200m*l* separating funnels, add water to bring each of the volumes to 150m*l*. Add the extraction solvent used in the sample pretreatment and conduct measurement using to the following conditions.

Measurement:

Measurement wavelength: 223.3nm

Calibration curve range : 0.5 – 5μg /m*l*

Measurement conditions :

Lamp current : 10mA

Slit width : 0.5nm

Lamp mode : BGC-D_2

Burner height : 7mm

Support gas : Air

Fuel gas flow rate: C_2H_2 0.8*l*/min (If flame appears reddish when sample is sprayed, decrease the amount of sample suctioned.)

- Cd

Reagent:

Cd standard solution (100μg of Cd/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to section b) is processed in the same way as that described in Procedure 1) for Bi.

2) For the standard solutions used to generate the calibration curve, take several aliquots of Cd standard solution (100μg of Cd/m*l*) with increasing concentration of Cd from 0 – 6μg, and to each of these,

accurately add 20m*l* of sulfuric acid (1+1) and 40m*l* of hydrochloric acid (1+1). Transfer these solutions to 200m*l* separating funnels, add water to bring each of the volumes to 150m*l*, add the extraction solvent used in the sample pretreatment and conduct measurement using the following conditions.

Measurement:

Measurement wavelength: 228.8nm

Calibration curve range : 0.05 – 0.4μg /m*l*

Measurement conditions :

Lamp current : 8mA

Slit width : 0.5nm

Lamp mode : BGC-D_2

Burner height : 7mm

Support gas : Air

Fuel gas flow rate: C_2H_2 0.8*l*/min (If flame appears reddish when sample is sprayed, decrease the amount of sample suctioned.)

- Pb I (Tri-n-octylmethylammonium bromide extraction)

Reagent:

Pb standard solution (100μg of Pb/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to section c) is processed in the same way as that described in Procedure 1) for Bi.
2) For the standard solutions used to generate the calibration curve, take several aliquots of Bi standard solution (100μg of Pb/m*l*) with increasing concentrations of Pb from 0 – 50μg, and to each of these, accurately add 15m*l* of sulfuric acid (1+1) and 30m*l* of hydrobromic acid (1+8). Transfer these solutions to 200m*l* separating funnels. Add water to bring each of the volumes to 150m*l*, add the extraction solvent used in the sample pretreatment and conduct measurement using the following conditions.

Measurement:

Measurement wavelength: 217.0nm

Calibration curve range : 0.5 – 5μg /m*l*

Measurement conditions :

Lamp current : 12mA

Slit width : 0.5nm

Lamp mode : BGC-D_2

Burner height : 7mm

Support gas : Air

Fuel gas flow rate: C_2H_2 0.8*l*/min (If flame appears reddish when sample is sprayed, decrease the amount of sample suctioned.)

- Pb II (Iron hydroxide precipitation separation)

Reagent:

Pb standard solution (100μg of Pb/m*l*): Same as for Pb I Procedure:

Procedure:

1) The sample solution pretreated according to section d) is processed in the same way as that described in Procedure 1) for Bi.
2) For the standard solutions used to generate the calibration curve, transfer to 100m*l* beakers several aliquots of Pb standard solution (100μg of Pb/m*l*) with increasing concentrations of Pb from 0 – 200μg. To each of these, accurately add 5m*l* of ferric ammonium sulfate (10 w/v%) and 2m*l* of sulfuric acid (1+1). Heat until evaporated, and add 10m*l* of hydrochloric acid (1+1), and use this for measurement.

Measurement:

Measurement wavelength: 283.3nm

Calibration curve range : 1 – 10μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 27)

- Zn

Reagent:

Zn standard solution (100μg of Zn/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to section b) is processed in the same way as that described in Procedure 1) for Bi.
2) For the standard solutions used to generate the calibration curve, follow the same procedure as that for Cd, using the Zn standard solution (100μg of Zn/m*l*) and the range of 0 – 3μg of Zn.

Measurement:

Measurement wavelength: 213.9nm

Calibration curve range : 0.05 – 0.3μg /m*l*

Measurement conditions :

Lamp current : 8mA

Slit width : 0.5nm

Lamp mode : BGC-D_2

Burner height : 7mm

Support gas : Air

Fuel gas flow rate: C_2H_2 0.8*l*/min (If flame appears reddish when sample is sprayed, decrease the amount of sample suctioned.)

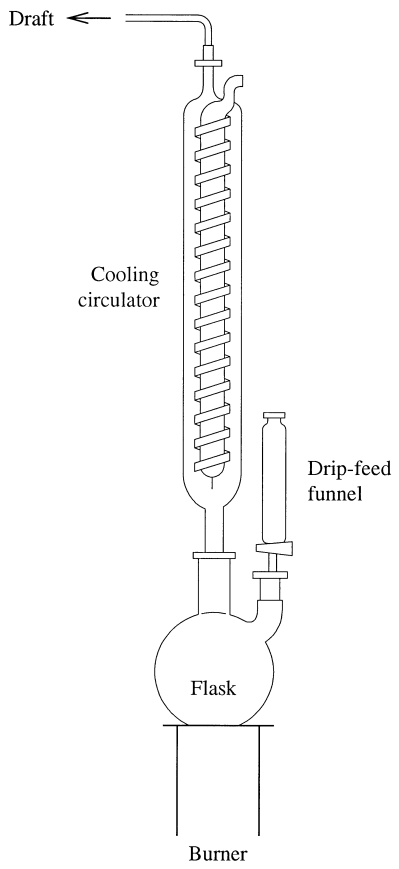
15.2.3 Reduction Vaporization Atomic Absorption Method

a) Target Element and quantitation range

Hg 0.00001% or higher

b) Sample pretreatment

Weigh out 3g of sample to the nearest 10mg and transfer to a 250 – 300m*l* decomposition flask, as shown in the figure at under. Attach the cooling circulator and drip-feed funnel. Add 20m*l* of acid mixture (sulfuric acid 3 parts + nitric acid 1 part + water 6 parts), and heat gently to decompose. After cooling, add 10m*l* of urea solution (5 w/v%), and boil for about 5 minutes. After allowing to cool a little, add about 30m*l* of water and add 5m*l* of potassium permanganate solution (5 w/v%), and boil for 20 minutes. After cooling, rinse the cooling circulator and drip-feed funnel with water. To this solution, drip-feed hydroxylammonium hydrochloride (20 w/v%). When the red color disappears due the permanganate ion, transfer to a 300m*l* reducing flask using water, and further dilute to bring up to 200m*l*.



Decomposition Flask with Cooling Circulator

c) Measurement procedure

Reagents:

1) Hg standard solution (0.1μg Hg/m*l*) : Refer to Cookbook Section 2, item 3 Preparing Standards
2) Stannous chloride solution (10 w/v%) : Add 10g of stannous chloride to 60m*l* of sulfuric acid (sulfuric acid to water ratio 1:20), and while heating, mix to dissolve. After cooling, bring up to 100m*l* using water.

Procedure:

1) The pretreated sample may be measured as it is.
2) For the calibration curve, prepare standard solutions starting with incremental volumes between 2.0 – 10.0m*l* of the Hg standard solution (0.1μg Hg/m*l*). To each, add 5m*l* of sulfuric acid (1+1), and bring up to 200m*l* using water.

Measurement:

Connect the MVU-1A Mercury Vaporizer Unit to the atomic absorption spectrophotometer, and measure the sample. For details on operation of the MVU-1A, refer to its accompanying documentation.

Measurement wavelength: 253.7nm

Calibration curve range : 1 – 5 ng/m*l*

Measurement conditions :

Lamp current : 4mA

Slit width : 0.5nm

Lamp mode : BGC-D_2

15.3 Electrolytic Copper Ore Analysis Method

Reference materials:

Japan Industrial Standard, Electrolytic Copper Ore Analysis Method JIS H 1291

15.3.1 Sample Pretreatment

a) As, Bi, Pb, Sb

1) Weigh out 10g of sample to the nearest 10mg and transfer to a 500m*l* beaker. Cover with a watch glass, add 80m*l* of nitric acid (1+1) to decompose, and boil gently to drive off the nitrogen oxide. After cooling to ambient temperature, rinse the bottom surface of the watch glass with water and remove the watch glass. Then add water to bring to a final volume of 200m*l*.

2) Add 4m*l* of iron (III) solution and 4m*l* of lanthanum nitrate, and while mixing this solution, add aqueous ammonia until the copper hydroxide precipitate dissolves. Then add 50m*l* more. Heat this solution and boil gently for 5 minutes. Filter out the precipitate using Type 5B filter paper, and rinse several times using warm ammonium rinse solution. Discard the rinse liquid and filtrate.

3) Using warm water, rinse the precipitate on the filter paper back into the original beaker, place the beaker under the funnel. Drip 10m*l* of a nitric acid mixture onto the filter paper to dissolve any remaining precipitate. Heat both the filtrate and rinse solution until the precipitate is completely dissolved and keep heating until the hydrogen peroxide is driven off. After cooling to ambient temperature, transfer to a 100m*l* volumetric flask using water, and bring up to volume with water.

Note:

1) Iron (III) solution: Add 20m*l* of nitric acid mixture to 0.5g of iron (99.9%), and after heating to dissolve, keep heating until hydrogen peroxide is driven off. After cooling to ambient temperature, dilute to 100m*l* using water (Fe 5μg/m*l*).

2) Lanthanum nitrate solution: Dissolve 1.6g of lanthanum nitrate 6-hydrate in 100m*l* of nitric acid (1+100) (La 5μg/m*l*).

3) Nitric acid mixture: Add 10m*l* of hydrogen peroxide to 100m*l* of nitric acid (1+1) (Prepare each time).

4) Ammonia rinse solution: Dissolve 15g of ammonium carbonate in 500m*l* of aqueous ammonia (2+50).

5) If there is more than 4μg of As, Bi, Pb and Sb in the sample, after completing step 1) of the above procedure, us a portion containing 2 – 4μg for steps 2) and thereafter.

b) Bi, Pb, Sb (Flame Atomic Absorption Method)

1) Weigh out 20g of sample to the nearest 10mg and transfer to a 500m*l* beaker. Cover with a watch glass, add 160m*l* of nitric acid to dissolve. Heat and boil gently to drive off the nitrogen oxide. After cooling to ambient temperature, rinse the bottom of the watch glass with water, remove the watch glass, and add water to bring volume to about 200m*l*.

2) Add 4m*l* of iron (III) solution and 4m*l* of lanthanum nitrate. While mixing this solution, add aqueous ammonia until the copper hydroxide precipitate dissolves, and then add 50m*l* more. Heat this solution and boil gently for 5 minutes. Filter out the precipitate using Type 5B filter paper, and rinse several times using warm ammonium rinse solution. Discard the rinse liquid and filtrate.

3) Using warm water, rinse the precipitate on the filter paper back into the original beaker, place the beaker under the funnel, and drip 10m*l* of a nitric acid mixture onto the filter paper to dissolve any remaining precipitate. Heat both the filtrate and rinse solution until the precipitate is completely dissolved. Transfer to a 200m*l* beaker using a small amount of water, and heat this solution to concentrate it down to a volume of about 10m*l*. After cooling to ambient temperature, transfer to a 25m*l* volumetric flask using water, and bring up to volume with water.

Note:

1) Iron (III) solution
2) Lanthanum nitrate solution
3) Nitric acid mixture
4) Ammonia rinse solution

Preparation of 1) – 4) above is the same as described in a) Notes 1) – 4).

5) If there is more than 250μg of Bi, Pb and Sb in the sample, transfer from the 25m*l* flask of step 3) a volume providing 50 – 250μg of the elements to another 25m*l* volumetric flask. Add 4m*l* of the iron (III) solution, 4m*l* of the lanthanum nitrate and 5m*l* of nitric acid (1+1), and bring up to volume with water.

c) Fe (Flame Atomic Absorption Method)

1) Weigh out 20g of sample to the nearest 10mg and transfer to a 500m*l* beaker. Cover with a watch glass, add 160m*l* of nitric acid to dissolve. Heat and boil gently to drive off the nitrogen oxide. After cooling to ambient temperature, rinse the bottom of the watch glass with water and remove the watch glass, transfer to a 200m*l* volumetric flask using water, and bring up to volume with water.

2) Transfer exactly 50m*l* of this solution to a beaker (If there is a large amount of Fe contained in the sample, decrease the volume so that 10 – 50μg of Fe is transferred). Heat until the solution is concentrated to a syrupy consistency. After allowing to cool, add 20m*l* of hydrochloric acid (1+1), heat gently to dissolve the salts, and cool to ambient temperature. Transfer to a 100m*l* separating funnel using hydrochloric acid (1+1), and add hydrochloric acid (1+1) to bring up to a volume of 50m*l*. Add 20m*l* of 4-methyl-2-pentanone isopentyl acetate solvent mixture, and shake vigorously. After allowing to separate into 2 layers, discard the aqueous layer. Repeat this procedure once more.

3) Add about 20m*l* of water to the organic phase, shake vigorously for about 1 minute, and after allowing to separate into 2 layers, transfer the aqueous phase to a 50m*l* beaker. Once again, add 5m*l* of water to the organic phase, shake vigorously for about 1 minute, and after allowing to separate into 2 layers, combine the aqueous phase with the previous portion in the 50m*l* beaker. Add 5m*l* of sulfuric acid (1+1) to this solution, and heat until evaporated. After allowing to cool, add 5m*l* of hydrochloric acid (1+1), and heat to dissolve the salts. After cooling to ambient temperature, transfer to a 25m*l* volumetric flask using water, and bring up to volume.

Note: 4-methyl-2-pentanone isopentyl acetate solvent mixture: Mix 1 part 4-methyl-2-pentanone with 1 part isopentyl acetate.

15.3.2 Electrical Heating Atomic Absorption Method

a) Target element and quantitation range

Element	Percent contained (%)
As	0.00005 – 0.005
Bi	0.00005 – 0.005
Pb	0.00005 – 0.005
Sb	0.00005 – 0.005

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current and slit width, refer to Cookbook Section 4, Item 7.5 Element Specific Measurement Conditions.

- As (Iron hydroxide - lanthanum hydroxide precipitation separation)

  Reagents:

  1) As standard solution (1μg of As/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
  2) Iron (III) solution
  3) Lanthanum nitrate solution

     Preparation of 2), 3) above is the same as described in a) Note 1), 2).

  Procedures:

  1) The sample solution pretreated according to step a) is measured just as it is. For blank measurement, perform the same pretreatment procedure on the reagent containing no sample, measure this solution, and use the value obtained to correct the value obtained in sample measurement.
  2) For the standard solutions to be used for generating the calibration curve, transfer both 4m*l* of the iron (III) solution and 4m*l* of the lanthanum nitrate solution to several 100m*l* volumetric flasks, and add increasing volumes of the As standard solution (1μg of As/m*l*) from 0 – 4.0m*l* (As content from 0 – 4μg). After adding 10m*l* of nitric acid (1+1) to each of the flasks, bring up to volume with water.

  Measurement:

  Measurement wavelength: 193.7nm
  Calibration curve range : 2 – 40 ng/m*l*
  Tube : High density graphite tube
  Sample injection volume : 20μ*l*

Heating conditions:

	TEMP (Ž)	TIME (sec)	HEAT	GAS (*l*/min)
STAGE 1	120	20	R	0.2
2	250	10	R	0.2
3	700	10	R	1.0
4	700	10	R	1.0
5	700	3	S	0.0H
6	2300	3	S	0.0H
7	2700	2	S	1.0

- Bi (Iron hydroxide - lanthanum hydroxide precipitation separation)

  Reagents:

  1) Bi standard solution (1μg of Bi/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
  2) Iron (III) solution
  3) Lanthanum nitrate solution

     Preparation of 2), 3) above is the same as described in a) Note 1), 2).

  Procedures:

  1) Sample measurement is the same as that described for As Procedures 1).
  2) For the standard solutions to be used for generating the calibration curve, transfer both 4m*l* of the iron (III) solution and 4m*l* of the lanthanum nitrate solution to several 100m*l* volumetric flasks, and add increasing volumes of the Bi standard solution (1μg of Bi/m*l*) from 0 – 4.0m*l* (Bi content from 0 – 4μg). After adding 10m*l* of nitric acid (1+1) to each of the flasks, bring up to volume with water.

  Measurement:

  Measurement wavelength: 223.1nm
  Calibration curve range : 2 – 40 ng/m*l*
  Tube : High density graphite tube
  Sample injection volume : 20μ*l*

Heating conditions:

	TEMP (Ž)	TIME (sec)	HEAT	GAS (*l*/min)
STAGE 1	120	20	R	0.2
2	250	10	R	0.2
3	600	10	R	1.0
4	600	15	S	1.0
5	600	3	S	0.0H
6	2000	3	S	0.0H
7	2700	2	S	1.0

- Pb (Iron hydroxide - lanthanum hydroxide precipitation separation)

  Reagents:

  1) Pb standard solution (1μg of Pb/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
  2) Iron (III) solution
  3) Lanthanum nitrate solution

     Preparation of 2), 3) above is the same as described in a) Note 1), 2).

  Procedures:

  1) Sample measurement is the same as that described for As Procedures 1).
  2) For the standard solutions to be used for generating the calibration curve, transfer both 4m*l* of the iron (III) solution and 4m*l* of the lanthanum nitrate solution to several 100m*l* volumetric flasks, and add increasing volumes of the Pb standard solution (1μg of Pb/m*l*) from 0 – 4.0m*l* (Pb content from 0 – 4μg). After adding 10m*l* of nitric acid (1+1) to each of the flasks, bring up to volume with water.

  Measurement:

  Measurement wavelength: 283.3nm
  Calibration curve range : 2 – 40 ng/m*l*
  Tube : High density graphite tube
  Sample injection volume : 20μ*l*

Heating conditions:

	TEMP (Ž)	TIME (sec)	HEAT	GAS (*l*/min)
STAGE 1	120	20	R	0.2
2	250	10	R	0.2
3	600	10	R	1.0
4	600	15	S	1.0
5	600	3	S	0.0H
6	2000	3	S	0.0H
7	2700	2	S	1.0

- Sb (Iron hydroxide - lanthanum hydroxide precipitation separation)

  Reagents:

  1) Sb standard solution (1μg of Sb/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
  2) Iron (III) solution
  3) Lanthanum nitrate solution
  4) Nitric acid mixture
  5) Ammonia rinse solution

     Preparation of 2) – 5) above is the same as described in a) Note 1) – 4).

  Procedures:

  1) Sample measurement is the same as that described for As Procedures 1).
  2) For the standard solutions to be used for generating the calibration curve, transfer 0 – 4.0m*l* of Sb standard solution (1μg of Sb/m*l*) in increasing concentrations (containing 0 – 4μg of Sb) to several beakers. After adding 60m*l* of nitric acid (1+1) to each beaker, add water to bring total volume to about 200m*l*. Add both 4m*l* of the iron (III) solution and 4m*l* of the lanthanum nitrate solution to these beakers. While mixing, add 160m*l* of aqueous ammonia. Heat this solution and boil gently for 5 minutes. Filter out the precipitate using Type 5B filter paper, and rinse several times using warm ammonium rinse solution. Discard the rinse liquid and filtrate.

Using warm water, rinse the precipitate on the filter paper back into the original beaker. Place the beaker under the funnel, and drip 10m*l* of the nitric acid mixture onto the filter paper to dissolve any remaining precipitate. Rinse the filter paper thoroughly using warm nitric acid (1+50). Heat both the filtrate and rinse solution until the precipitate is completely dissolved, then transfer the solution to a 200m*l* beaker using a small amount of water. Heat again to concentrate, and after cooling to ambient temperature, transfer to a 100m*l* volumetric flask using water, and bring up to volume with water. Use this solution for measurement.

Measurement:

Measurement wavelength: 217.6nm
Calibration curve range : 2 – 40 ng/m*l*
Tube : High density graphite tube
Sample injection volume : 20μ*l*
Heating conditions :

	TEMP (Ž)	TIME (sec)	HEAT	GAS (*l*/min)
STAGE 1	120	20	R	0.2
2	250	10	R	0.2
3	600	10	R	1.0
4	600	15	S	1.0
5	600	3	S	0.0H
6	2200	3	S	0.0H
7	2700	2	S	1.0

15.3.3 Flame Atomic Absorption Method

a) Target element and quantitation range

Element	Percent contained (%)
Bi	0.0001 – 0.01
Fe	0.0001 – 0.01

Pb	0.0001 – 0.01
Sb	0.0001 – 0.01

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current, slit width and flame conditions, refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions.

- Bi

Reagents:

1) Bi standard solution (50μg of Bi/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Iron (III) solution
3) Lanthanum nitrate solution

Preparation of 2), 3) above is the same as described in a) Note 1), 2).

Procedures:

1) The sample solution pretreated according to step b) is measured just as it is. For blank measurement, perform the same pretreatment procedure on the reagent containing no sample. Measure this solution, and use the value obtained to correct the value obtained in sample measurement.
2) For the standard solutions to be used for generating the calibration curve, transfer both 4m*l* of the iron (III) solution and 4m*l* of the lanthanum nitrate solution to several 25m*l* volumetric flasks, and add increasing volumes of the Bi standard solution (50μg of Bi/m*l*) from 0 – 5.0m*l* (Bi content from 0 – 250μg). After adding 2m*l* of nitric acid (1+1) to each of the flasks, bring up to volume with water.

Measurement:

Measurement wavelength: 223.1nm

Calibration curve range : 1 – 10μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 8)

- Fe

Reagents:

Fe standard solution (10μg of Fe/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.

Procedure:

1) The sample solution pretreated according to step c) is measured just as it is. For blank measurement, perform the same pretreatment procedure on the reagent containing no sample, measure this solution, and use the value obtained to correct the value obtained in sample measurement.
2) For the standard solutions to be used for generating the calibration curve, transfer increasing volumes of the Fe standard solution (10μg of Fe/m*l*) from 0 – 5.0m*l* (Fe content from 0 – 50μg) to several 25m*l* volumetric flasks. After adding 50m*l* of hydrochloric acid (1+1) to each of the flasks, bring up to volume with water.

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.2 – 2μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

- Pb

Reagents:

1) Pb standard solution (50μg of Pb/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Iron (III) solution
3) Lanthanum nitrate solution

Preparation of 2), 3) above is the same as described in a) Note 1), 2).

Procedures:

1) Sample measurement is conducted using the same procedure as that described for Bi, Procedure, 1).
2) For the standard solutions to be used in generating the calibration curve, transfer both 4m*l* of the iron (III) solution and 4m*l* of the lanthanum nitrate solution to several 25m*l* volumetric flasks. Add increasing volumes of the Pb standard solution (50μg of Pb/m*l*) from 0 – 5.0m*l* (Pb content from 0 – 250μg), and after adding 2m*l* of nitric acid (1+1) to each of the flasks, bring up to volume with water.

Measurement:

Measurement wavelength: 283.3nm

Calibration curve range : 1 – 10μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 27)

- Sb

Reagents:

1) Sb standard solution (50μg of Sb/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Iron (III) solution
3) Lanthanum nitrate solution
4) Nitric acid mixture
5) Ammonia rinse solution

Preparation of 2) – 5) above is the same as described in a) Note 1) – 4).

Procedures:

1) Sample measurement is the same as that described for Bi Procedure 1).
2) For the standard solutions to be used in generating the calibration curve, transfer 0 – 5.0m*l* of Sb standard solution (50μg of Sb/m*l*) in increasing concentrations (containing 0 – 250μg of Sb) to several 500m*l* beakers. After adding 60m*l* of nitric acid (1+1) to each beaker, add water to bring total volume to about 200m*l*. Add both 4m*l* of the iron (III) solution and 4m*l* of the lanthanum nitrate solution to these beakers. While mixing add 160m*l* of aqueous ammonia. Heat this solution and boil gently for 5 minutes. Filter out the precipitate using Type 5B filter paper, and rinse several times using warm ammonium rinse solution. Discard the rinse liquid and filtrate.

   Using warm water, rinse the precipitate on the filter paper back into the original beaker. Place the beaker under the funnel, and drip 10m*l* of the nitric acid mixture onto the filter paper to dissolve any remaining precipitate. Rinse the filter paper thoroughly using warm nitric acid (1+50). Heat both the filtrate and rinse solution until the precipitate is completely dissolved, and then transfer the solution to a 200m*l* beaker using a small amount of water. Heat again to concentrate, and after cooling to ambient temperature, transfer to a 25m*l* volumetric flask using water, and bring up to volume with water. Use this solution for measurement.

Measurement:

Measurement wavelength: 217.6nm

Calibration curve range : 1 – 10μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 32)

15.4 Aluminum and Aluminum Alloy Analysis Method

Reference materials:

Japan Industrial Standard, Aluminum and Aluminum Alloy Atomic Absorption Analysis Method JIS H 1306

15.4.1 Sample Pretreatment

a) Weighing out sample

The sample weight is based on the amount of the target element contained in the sample. The following table gives sample weights based on the percentage of target element in the sample.

Element	Percent contained (%)	Weight (g)
Bi	0.1 – 1.0	1.00
Cr	0.01 – 0.5	1.00
Cu	0.005 – 1.5	1.00
	1.5 – 5.0	0.20
Fe	0.005 – 1.5	1.00
Mn	0.005 – 1.5	1.00
Mg	0.005 – 1.5	1.00
	1.5 – 5.0	0.20
Ni	0.005 – 1.5	1.00
	1.5 – 3.0	0.20
Pb	0.1 – 1.0	1.00
Zn	0.005 – 1.5	1.00
	1.5 – 6.0	0.20

b) Preparing sample solution

Weigh out the sample and transfer it to a 300m*l* beaker. Cover with a watch glass, add 30m*l* of hydrochloric acid (1+1) and heat gently to decompose. To this solution add 1m*l* of hydrogen peroxide, and heat to completely decompose the sample. Cool to ambient temperature, and then rinse the bottom of the watch glass and inner wall of the beaker with water. Then remove the watch glass. Transfer the solution to a 100m*l* volumetric

flask, and add water to bring up volume.

Note:

1) For Cr, transfer the solution to a 200m*l* volumetric flask, add 1.5m*l* of ammonium hydrochloride solution (270g/*l*), and bring up to volume with water.
2) When the contained percentages of Cu, Zn, Mg and Ni are greater than 1.5%, transfer the solution containing the decomposed sample to a 200m*l* volumetric flask.
3) For samples that are difficult to decompose, add 2m*l* of tin solution or 2m*l* of nickel solution.
4) Tin solution: Weigh out 0.1g of tin (>99.9%), transfer to a 200m*l* beaker, add 30m*l* of hydrochloric acid (1+1). While placing in contact with platinum, heat at 50 – 80°C to decompose. Then cool to ambient temperature, and add hydrochloric acid to bring up to 100m*l*.
5) Nickel solution: Weigh out 0.1g of nickel (>99.9%), transfer to a 200m*l* beaker, add 10m*l* of nitric acid (1+1), and heat gently to decompose. Cool to ambient temperature and add water to bring volume to 100m*l*.

15.4.2 Flame Atomic Absorption Method

a) Target element and quantitation range

Element	Percent contained (%)
Bi	0.1 – 1.0
Cr	0.01 – 0.5
Cu	0.005 – 5.0
Fe	0.005 – 1.5
Mn	0.005 – 1.5
Mg	0.005 – 5.0
Ni	0.005 – 3.0
Pb	0.1 – 1.0
Zn	0.005 – 6.0

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current, slit width and flame conditions, refer to Cookbook Section 3, Item 6.4

Element Specific Measurement Conditions.

- Bi

  Reagents:

  Bi standard solution (1000μg of Bi/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.

  Procedure:

  1) The sample solution pretreated according to step b) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Bi standard solution. The value obtained can be used to correct the value obtained in sample measurement.
  2) For the standard solutions to be used in generating the calibration curve, measure out 1.0gram of aluminum (as pure as possible, containing no Bi or containing a known quantity of Bi) for each solution, and transfer these to several 300m*l* beakers. To these beakers, transfer increasing volumes of Bi standard solution (1000μg of Bi/m*l*) from 0 – 10.0m*l* (containing 0 – 10μg of Bi). The remainder of the procedure is the same as that described for Sample Pretreatment b) Preparing sample solutions.

  Measurement:

  Measurement wavelength: 223.1nm

  Calibration curve range : 1 – 100μg /m*l*

  Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 8)

  Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Cr

  Reagents:

  1) Cr standard solution (1000μg of Cr/m*l*)
  2) Cr standard solution (100μg of Cr/m*l*)

     For Preparation of 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards.

Procedure:

1) The pretreated sample solution can be measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cr standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, measure out 1.0gram of aluminum (as pure as possible, containing no Cr or containing a known quantity of Cr) for each solution, and transfer these to several 300m*l* beakers. Depending on the amount of Cr contained in the sample, Cr standard solution is added to the beakers in increasing concentrations as indicated in the table below. The remainder of the procedure is the same as that described for Sample Pretreatment b) Preparing sample solutions.

Percent Cr in Sample (%)	Cr standard used	Amt of Cr added (m*l*)
0.01 – 0.1	2) 100μg of Cr/m*l*	0 – 10.0
>0.1	1) 1000μg of Cr/m*l*	0 – 5.0

Measurement:

Measurement wavelength: 357.9nm

Calibration curve range : 0.3 – 25μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 13)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Cu

Reagents:

1) Cu standard solution (1000μg of Cu/m*l*)
2) Cu standard solution (100μg of Cu/m*l*)

For Preparation of 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards.

Procedure:

1) When the pretreated sample solution contains less than 0.1 % of Cu, it can be measured just as it is. However, if the pretreated sample solution contains more than 0.1%, use an aliquot indicated in the following table for measurement.

Percent Cu in Sample (%)	Aliquot (m*l*)	Volumetric flask
0.1 – 0.5	50	100
0.5 – 1.5	20	100
1.5 – 5.0	20	200

For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cu standard solution is prepared and measured, and the value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, measure out aluminum (as pure as possible, containing no Cu or containing a known quantity of Cu) to the same weight within 10mg of the sample pretreated according to step a) of the pretreatment description for sample. Transfer this quantity to several 300m*l* beakers. Depending on the amount of Cu contained in the sample, Cu standard solution is added to the beakers in increasing concentrations as indicated in the table below. The remainder of the procedure is the same as that described for Sample Pretreatment b) Preparing sample solutions.

Percent Cu in Sample (%)	Cu standard used	Amt of Cu added (m*l*)
0.005 – 0.1	2) 100μg of Cu/m*l*	0 – 10.0
0.1 – 0.5	1) 1000μg of Cu/m*l*	0 – 5.0
0.5 – 1.5	1) 1000μg of Cu/m*l*	0 – 15.0
1.5 – 5.0	1) 1000μg of Cu/m*l*	0 – 10.0

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.2 – 30μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 15)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Fe

Reagents:

1) Fe standard solution (1000μg of Fe/m*l*)
2) Fe standard solution (100μg of Fe/m*l*)

   For Preparation of 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards.

Procedure:

1) When the pretreated sample solution contains less than 0.1% of Fe, it can be measured just as it is. However, if the pretreated sample solution contains more than 0.1%, use an aliquot indicated in the following table for measurement.

Percent Cu in Sample (%)	Aliquot (m*l*)	Volumetric flask
0.1 – 0.5	50	100
0.5 – 1.5	20	100

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Fe standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, measure out 1.0gram of aluminum (as pure as possible, containing no Fe or containing a known quantity of Fe) for each solution, and transfer these to several 300m*l* beakers. Depending on the amount of Fe contained in the sample, Fe standard solution is

added to the beakers in increasing concentrations as indicated in the table below. The remainder of the procedure is the same as that described for Sample Pretreatment b) Preparing sample solutions.

Percent Fe in Sample (%)	Fe standard used	Amt of Fe added (m*l*)
0.005 – 0.1	2) 100μg of Fe/m*l*	0 – 10.0
0.1 – 0.5	1) 1000μg of Fe/m*l*	0 – 5.0
0.5 – 1.5	1) 1000μg of Fe/m*l*	0 – 15.0

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.3 – 30μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Mn

Reagents:

1) Mn standard solution (1000μg of Mn/m*l*)
2) Mn standard solution (100μg of Mn/m*l*)

For Preparation of 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards.

Procedure:

1) When the pretreated sample solution contains less than 0.1% of Mn, it can be measured just as it is. However, if the pretreated sample solution contains more than 0.1%, use an aliquot indicated in the following table for measurement.

Percent Mn in Sample (%)	Aliquot (m*l*)	Volumetric flask
0.1 – 0.5	50	100
0.5 – 1.5	20	100

For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Mn standard solution is prepared and measured, and the value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, measure out 1.0gram of aluminum (as pure as possible, containing no Mn or containing a known quantity of Mn) for each solution, and transfer these to several 300m*l* beakers. Depending on the amount of Mn contained in the sample, Mn standard solution is added to the beakers in increasing concentrations as indicated in the table below. The remainder of the procedure is the same as that described for Sample Pretreatment b) Preparing sample solutions.

Percent Mn in Sample (%)	Mn standard used	Amt of Mn added (m*l*)
0.005 – 0.1	2) 100μg of Mn/m*l*	0 – 10.0
0.1 – 0.5	1) 1000μg of Mn/m*l*	0 – 5.0
0.5 – 1.5	1) 1000μg of Mn/m*l*	0 – 15.0

Measurement:

Measurement wavelength: 279.5nm

Calibration curve range : 0.2 – 30μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 22)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Mg

Reagents:

1) Mg standard solution (1000μg of Mg/m*l*)
2) Mg standard solution (100μg of Mg/m*l*)

For Preparation of 1) and 2), refer to Cookbook Section 2, Item 3

Preparing Standards.

3) Strontium solution: Dissolve 30.8g of strontium chloride 6-hydrate in water, and add water to bring solution up to 100m*l*.

Procedure:

1) Prepare an aliquot from the pretreated sample solution according to the following table, and use that aliquot for measurement.

Percent Mg in Sample (%)	Aliquot (m*l*)	Amt of Sr Added (m*l*)	Volumetric flask
0.005 – 0.1	20.0	2.0	100
0.1 – 0.5	10.0	2.0	100
0.5 – 1.5	5.0	2.0	100
1.5 – 5.0	5.0	2.0	100

For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Mg standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, measure out aluminum (as pure as possible, containing no Mg or containing a known quantity of Mg) to the same weight within 10mg of the sample pretreated according to step a) of the pretreatment description for sample. Transfer this quantity to several 300m*l* beakers. Depending on the amount of Mg contained in the sample, Mg standard solution is added to the beakers in increasing concentrations as indicated in the table below. The remainder of the procedure is the same as that described for Sample Pretreatment b) Preparing sample solutions.

Percent Mg in Sample (%)	Mg standard used	Amt of Mg added (m*l*)
0.005 – 0.1	2) 100μg of Mg/m*l*	0 – 10.0
0.1 – 0.5	1) 1000μg of Mg /m*l*	0 – 5.0
0.5 – 1.5	1) 1000μg of Mg /m*l*	0 – 15.0
1.5 – 5.0	1) 1000μg of Mg /m*l*	0 – 10.0

Measurement:

Measurement wavelength: 285.2nm

Calibration curve range : 0.2 – 7.5μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 21)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Ni

Reagents:

1) Ni standard solution (1000μg of Ni /m*l*)
2) Ni standard solution (100μg of Ni /m*l*)

For Preparation of 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards.

Procedure:

1) When the pretreated sample solution contains less than 0.1% of Ni, it can be measured just as it is. However, if the pretreated sample solution contains more than 0.1%, use an aliquot indicated in the following table for measurement.

Percent Ni in Sample (%)	Aliquot (m*l*)	Volumetric flask
0.1 – 0.5	50	100
0.5 – 1.5	20	100
1.5 – 5.0	20	100

For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Ni standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, measure out aluminum (as pure as possible, containing no Ni or containing a known quantity of Ni) to the same weight within

10mg of the sample pretreated according to step a) of the pretreatment description for sample. Transfer this quantity to several 300m*l* beakers. Depending on the amount of Ni contained in the sample, Ni standard solution is added to the beakers in increasing concentrations as indicated in the table below. The remainder of the procedure is the same as that described for Sample Pretreatment b) Preparing sample solutions.

Percent Ni in Sample (%)	Ni standard used	Amt of Ni added (m*l*)
0.005 – 0.1	2) 100μg of Ni/m*l*	0 – 10.0
0.1 – 0.5	1) 1000μg of Ni/m*l*	0 – 5.0
0.5 – 1.5	1) 1000μg of Ni/m*l*	0 – 15.0
1.5 – 3.0	1) 1000μg of Ni/m*l*	0 – 10.0

Measurement:

Measurement wavelength: 232.0nm

Calibration curve range : 0.5 – 30μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 25)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Pb

Reagents:

Pb standard solution (1000μg of Pb/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.

Procedure:

1) Sample measurement is conducted as described for Bi, Procedure, Step 1).
2) For the standard solutions to be used in generating the calibration curve, measure out 1.0gram of aluminum (as pure as possible, containing no Pb or containing a known quantity of Pb) for each solution, and transfer these to several 300m*l* beakers. To these

beakers, transfer increasing volumes of Pb standard solution (1000μg of Pb/m*l*) from 0 – 10.0m*l* (containing 0 – 10mg of Pb). The remainder of the procedure is the same as that described for Sample Pretreatment b) Preparing sample solutions.

Measurement:

Measurement wavelength: 283.3nm

Calibration curve range : 1 – 100μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 27)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Zn

Reagents:

1) Zn standard solution (1000μg of Zn/m*l*)
2) Zn standard solution (100μg of Zn/m*l*)

For Preparation of 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards.

Procedure:

1) For measurement, use an aliquot of the pretreated sample as indicated in the following table.

Percent Zn in Sample (%)	Aliquot (m*l*)	Volumetric flask
0.005 – 0.1	50.0	100
0.1 – 0.5	20.0	100
0.5 – 1.5	5.0	100
1.5 – 6.0	10.0	100

For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Zn standard solution is prepared and measured, and the value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration

curve, measure out aluminum (as pure as possible, containing no Zn or containing a known quantity of Zn) to the same weight within 10mg of the sample pretreated according to step a) of the pretreatment description for sample. Transfer this quantity to several 300m*l* beakers. Depending on the amount of Zn contained in the sample, Zn standard solution is added to the beakers in increasing concentrations as indicated in the table below. The remainder of the procedure is the same as that described for Sample Pretreatment b) Preparing sample solutions.

Percent Zn in Sample (%)	Zn standard used	Amt of Zn added (m*l*)
0.005 – 0.1	2) 100μg of Zn/m*l*	0 – 10.0
0.1 – 0.5	1) 1000μg of Zn/m*l*	0 – 5.0
0.5 – 1.5	1) 1000μg of Ni/m*l*	0 – 15.0
1.5 – 6.0	1) 1000μg of Ni/m*l*	0 – 12.0

Measurement:

Measurement wavelength: 213.9nm

Calibration curve range : 0.1 – 10μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 44)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

15.5 Diecast Zinc Alloy Analysis Method

Reference materials:

Japan Industrial Standard, Diecast Zinc Alloy Analysis Method JIS H 1551

15.5.1 Sample Pretreatment

a) Al, Cd, Cu, Fe Mg, Pb

Weigh out 5.0g of sample to the nearest 1mg, and transfer to a 300m*l* beaker. Cover with a watch glass, and add 30m*l* of the acid mixture (45 parts hydrochloric acid, 1 part nitric acid) to decompose. After the intense reaction subsides, keep heating gently until decomposition is complete and the total volume has decreased to about 25m*l*. After cooling to ambient temperature, rinse the bottom of the watch glass with water and remove the watch glass. After adding about 20m*l* of water and 20m*l* of hydrochloric acid (1+1), transfer the solution to a 100m*l* volumetric flask and bring up to volume with water.

b) Sn

Weigh out 1.0g of sample to the nearest 10mg, and transfer to a 300m*l* beaker. Cover with a watch glass, and add 20m*l* of nitric (1+2), and after the intense reaction subsides, keep heating gently until decomposition is complete. After cooling to ambient temperature, rinse the bottom of the watch glass with water and remove the watch glass. Transfer the solution to a 100m*l* volumetric flask and bring up to volume with water.

15.5.2 Electrical Heating Atomic Absorption Method

a) Target element and quantitation range

Element	Percent Contained (%)
Sn	0.0001 – 0.004

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current and slit width, refer to Cookbook Section 4, Item 7.5 Element Specific Measurement Conditions.

- Sn

Reagents:

1) Sn standard solution (2μg of Sn/m*l*)

2) Cu standard solution (1μg of Cu/m*l*)

For 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards

3) Zn: >99.99%, containing <0.00005% Sn

4) Aluminum solution (2μg Al/m*l*): Weigh out 0.500g of aluminum (>99.9%, containing <0.0025% Sn), transfer to a 300m*l* beaker, add 20m*l* of nitric acid (1+2) and 2m*l* of nickel solution (1g of nickel nitrate 6-hydrate dissolved in 250m*l* of water) to decompose. After cooling to ambient temperature, transfer solution to a 250m*l* volumetric flask using water, and bring up to volume with water.

Procedure:

1) The sample solution pretreated according to step b) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Sn standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, weigh out 1.0gram of Zn into each of several 300m*l* beakers, and add 20m*l* of nitric acid (1+2) to decompose. After cooling to ambient temperature, transfer each of these solutions to a 100m*l* volumetric flask using water. Then add 20m*l* of aluminum solution (2mg of Al/m*l*) and 10m*l* of Cu standard solution (1mg of Cu/m*l*). To these, add increasing volumes of Sn standard solution (2μg of Sn/m*l*) from 0 – 20.0m*l* (containing 0 – 40μg of Sn). Finally, bring up to volume with water.

Note: If the sample contains less than 0.25% Cu, do not add the Cu standard solution.

Measurement:

Measurement wavelength: 286.3nm

Calibration curve range : 10 – 400 ng/m*l*

Tube : Pyrolized graphite tube

Sample injection volume : 10μ*l*

Heating conditions :

	TEMP (Ž)	TIME (sec)	HEAT	GAS (*l*/min)
STAGE 1	120	15	R	0.2
2	250	10	R	0.2
3	500	10	R	1.0
4	500	10	R	1.0
5	500	3	S	0.0
6	2400	3	S	0.0
7	2800	2	S	1.0

15.5.3 Flame Atomic Absorption Method

a) Target element and quantitation range

Element	Percent Contained (%)
Al	3.0 – 5.0
Cd	0.0001 – 0.010
Cu	0.005 – 2.0
Fe	0.0002 – 0.20
Mg	0.002 – 0.10
Pb	0.001 – 0.010

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current, slit width and flame conditions, refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions.

- Al

Reagents:

1) Al standard solution (1g of Al/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Zn standard solution (25mg of Zn/m*l*): Weigh out 25.0g of zinc (>99.995%), transfer to a 500m*l* beaker, add 100m*l* of hydrochloric

acid (1+1) to decompose, and heat to concentrate to a syrupy consistency. After cooling to room temperature, add about 200m*l* of water to dissolve the salts. Transfer the solution to a 1000m*l* volumetric flask using water, and bring up to volume with water.

Procedure:

1) Accurately transfer 5m*l* of sample solution pretreated according to step a) to a 250m*l* volumetric flask, add 50m*l* of hydrochloric acid (1+1), and bring up to volume with water.

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Al standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, transfer 10m*l* of zinc solution (25mg of Zn/m*l*) to each of several 250m*l* volumetric flasks, and add 50m*l* of hydrochloric acid (1+1) to each flask. Add Al standard solution (1mg of Al/m*l*) in increasing volumes from 0 – 15.0m*l* (containing 0 – 150m*l* of Al), and bring the solutions up to volume with water.

Measurement:

Measurement wavelength: 309.3nm

Calibration curve range : 10 – 60μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 2)

- Cd

Reagents:

1) Cd standard solution (20mg of Cd/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.

2) Zn standard solution (192μg of Zn/m*l*): Weigh out 96g of zinc (>99.995%), transfer to a 1000m*l* beaker, add 400m*l* of hydrochloric acid (1+1) to decompose. Heat to concentrate to a syrupy consistency. After cooling to room temperature, add about 400m*l* of water to dissolve the salts, and transfer the solution to a 500m*l* volumetric. Add about 20g of powdered zinc (JIS K 8013) and set aside for about 30 minutes, occasionally shaking the flask, and then bring up to volume with water. Filter this solution through type 5B dry filter

paper into a container which can be sealed.

3) Aluminum solution (20mg of Al/m*l*): weigh out 2.0g aluminum (>99.9%, containing <0.0002% of cadmium), transfer to a 300m*l* beaker, add 40m*l* of hydrochloric acid (1+1) and 2m*l* of nickel solution (1g of nickel chloride) dissolved in 250m*l* of water), add 1 – 2m*l* of nitric acid, and heat to dissolve the remaining undissolved aluminum. Then continue heating to drive off the hydrogen peroxide. After cooling to ambient temperature, transfer the solution to 100m*l* volumetric flask using water, and bring up to volume with water.

Procedure:

1) The sample solution pretreated according to step a) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cd standard solution is prepared and measured, and the value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, transfer 25m*l* of Zn standard solution (192mg of Zn/m*l*): and 10m*l* of aluminum solution (20mg of Al/m*l*) to each of several 100m*l* volumetric flasks, and add 20m*l* of hydrochloric acid (1+1) to each flask. Add Cd standard solution (20μg of Cd/m*l*) in increasing volumes from 0 – 25.0m*l* (containing 0 – 500μg of Cd), and bring the solutions up to volume with water.

Measurement:

Measurement wavelength: 228.8nm

Calibration curve range : 0.05 – 5μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4,11)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Cu

Reagents:

1) Cu standard solution (250μg of Cu/m*l*): refer to Cookbook Section 2,

Item 3 Preparing Standards.

2) Zn solution (192mg of Zn/m*l*): Same procedure as that for Cd Reagent, 2)
3) Zn solution (25mg of Zn/m*l*): Same procedure as that for Al Reagent, 2)
4) Aluminum solution (20mg of Al/m*l*): Same procedure as that for Cd Reagent, 3)

Procedure:

1) When the sample solution pretreated according to step a) contains from 0.005 – 0.05% Cu, it is measured just as it is. However, if the amount of Cu in the sample is 0.05 – 2.0%, accurately transfer 5m*l* of the sample solution to a 250m*l* volumetric flask, add 50m*l* of hydrochloric acid (1+1), and bring up to volume with water. Use this solution for measurement.

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cu standard solution is prepared and measured, and the value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, when the sample contains from 0.005 – 0.05% Cu, transfer 25m*l* of Zn standard solution (192mg of Zn/m*l*): and 10m*l* of aluminum solution (20mg of Al/m*l*) to each of several 100m*l* volumetric flasks, and add 20m*l* of hydrochloric acid (1+1) to each flask. Add Cu standard solution (250μg of Cu/m*l*) in increasing volumes from 0 – 10.0m*l* (containing 0 – 2.5μg of Cd), and bring the solutions up to volume with water.

   When the sample contains from 0.05 – 2.0% Cu, transfer 10m*l* of zinc solution (25mg of Zn/m*l*) to each of several 250m*l* volumetric flasks, and add 50m*l* of hydrochloric acid (1+1). Add Cu standard solution (250μg of Cu/m*l*) in incremental volumes from 0 – 20.0m*l* (containing 0 – 5μg of Cu) to the flasks, and bring up to volume with water.

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.5 – 25μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 15)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Fe

Reagents:

1) Fe standard solution (20μg of Fe/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Zn solution (192mg of Zn/m*l*):: Same procedure as that for Cd Reagent, 2)
3) Aluminum solution (20mg of Al/m*l*): Same procedure as that for Cd Reagent, 3)

Procedure:

1) When the sample solution pretreated according to step a) contains from 0.0002 – 0.016 % Fe, it is measured just as it is. However, if the amount of Fe in the sample is 0.016 – 0.20%, accurately transfer 5m*l* of the sample solution to a 100m*l* volumetric flask. Add 20m*l* of hydrochloric acid (1+1), and bring up to volume with water. Use this solution for measurement.

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Fe standard. The value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, when the sample contains from 0.0002 – 0.016% Fe, transfer 25m*l* of Zn standard solution (192mg of Zn/m*l*), and 10m*l* of aluminum solution (20mg of Al/m*l*) to each of several 100m*l* volumetric flasks. Add 20m*l* of hydrochloric acid (1+1) to each flask. Add Fe standard solution (20μg of Fe/m*l*) in increasing volumes from 0 – 40.0m*l* (containing 0 – 800μg of Fe), and bring the solutions up to volume with water.

   When the sample contains from 0.016 – 0.20% Fe, transfer 1.3m*l* of zinc solution (192mg of Zn/m*l*) and 0.5m*l* of aluminum solution

(20mg of Al/m*l*) to each of several 100m*l* volumetric flasks, and add 20m*l* of hydrochloric acid (1+1). Add Fe standard solution (20μg of Fe/m*l*) in incremental volumes from 0 – 30.0m*l* (containing 0 – 600μg of Fe) to the flasks, and bring up to volume with water.

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.1 – 8μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Mg

Reagents:

1) Mg standard solution (10μg of Mg/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Zn solution (25mg of Zn/m*l*): Same procedure as that for Al Reagent, 2)
3) Lanthanum solution (25mg of La/m*l*): Weigh out 29.5g of lanthanum oxide, transfer it to a 500m*l* beaker, gradually add 30m*l* of hydrochloric acid. After adding 200m*l* of water, heat until completely decomposed. Cool to ambient temperature, then transfer the solution to a 1000m*l* volumetric flask and bring up to volume with water.

Procedure:

1) Accurately transfer 250m*l* of sample solution pretreated according to step a) to a 250m*l* volumetric flask. Add 50m*l* of hydrochloric acid (1+1) and 25m*l* of lanthanum solution (25mg of La/m*l*), and bring up to volume with water.

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Mg standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, transfer 10m*l* of Zn solution (25mg of Zn/m*l*): to each of several 250m*l* volumetric flasks, and add 50m*l* of hydrochloric acid

(1+1) to each of the flasks. Add increasing volumes of Mg standard solution (10μg of Mg/m*l*) from 0 – 25.0m*l* (containing 0 – 250μg of Mg) to each of the flasks, add 25m*l* lanthanum solution (25mg of La/m*l*), and then bring up to volume with water.

Measurement:

Measurement wavelength: 285.2nm

Calibration curve range : 0.02 – 1μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 21)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Pb

Reagents:

1) Pb standard solution (20μg of Pb/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Zn solution (192mg of Zn/m*l*): Same procedure as that for Cd Reagent, 2)
3) Aluminum solution (20mg of Al/m*l*): Same procedure as that for Cd Reagent, 3)

Procedure:

1) The sample solution pretreated according to step a) is measured just as it is.

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Pb standard solution. The value obtained can be used to correct the value obtained in samplemeasurement.
2) For the standard solutions to be used in generating the calibration curve, transfer 25m*l* of Zn standard solution (192mg of Zn/m*l*), and 10m*l* of aluminum solution (20mg of Al/m*l*) to each of several 100m*l* volumetric flasks. Add 20m*l* of hydrochloric acid (1+1) to each flask. Add Pb standard solution (20μg of Pb/m*l*) in increasing volumes from 0 – 25.0m*l* (containing 0 – 500μg of Pb), and bring the solutions up to volume with water.

Measurement:

Measurement wavelength: 217.0nm

Calibration curve range : 0.5 – 5μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 26)

15.6 Zinc Alloy Analysis Method

Reference materials: Japan Industrial Standard:

Zinc Alloy Lead Quantitation Method	JIS H 1108
Zinc Alloy Iron Quantitation Method	JIS H 1109
Zinc Alloy Cadmium Quantitation Method	JIS H 1110

15.6.1 Sample Pretreatment

a) Cd, Fe, Pb

Weigh out 5.0g of sample and transfer to a 300m*l* beaker. Cover with a watch glass, and add 30m*l* of acid mixture (45 parts hydrochloric acid, 1 part nitric acid). After the intense reaction subsides, keep heating gently until decomposition is complete and the total volume has decreased to about 25m*l*. After cooling to ambient temperature, rinse the bottom of the watch glass with water and remove the watch glass. After adding about 20m*l* of water and 20m*l* of hydrochloric acid (1+1), transfer the solution to a 100m*l* volumetric flask and bring up to volume with water.

b) Pb (Iron hydroxide precipitation separation)

Weigh out 100g of sample, transfer to a 500m*l* beaker, and add 5m*l* of ferric ammonium sulfate (III). Cover with a watch glass, and add 60m*l* of acid mixture (45 parts hydrochloric acid, 1 part nitric acid). After the intense reaction subsides, keep heating gently until decomposition is complete and the total volume has decreased to about 25m*l*. After cooling to ambient temperature, rinse the bottom of the watch glass with water. Remove the watch glass, and add water to bring the volume to about 200m*l*.

While mixing this solution, add aqueous ammonia to dissolve the zinc hydroxide precipitate. After it dissolves, add 50m*l* more. Next add 15g of ammonium carbonate and heat. After boiling gently for about 5 minutes, set aside in a warm location (60 – 80°C) for 1 – 2 hours. Separate the precipitate using Type 5A filter paper, and rinse several times with warm ammonia rinse solution.

Transfer the precipitate on the filter paper to the original beaker by rinseing with warm water. Place the beaker under the filter, and drip 10m*l* of hydrochloric acid (1+1) onto the filter paper to dissolve the remaining precipitate on the filter paper and in the beaker. Rinse the filter paper thoroughly with warm hydrochloric acid (1+50).

Heat the combined filtrate and rinse solution to concentrate it to a volume of 10 – 20m*l*. After cooling, transfer to a 50m*l* volumetric flask using water, and bring up to volume with water.

Note: Ferric ammonium sulfate (III) solution: Dissolve 10g of ferric ammonium sulfate in 100m*l* of nitric acid (1+100). 1m*l* of this solution contains 12mg of Fe.

Ammonium rinse solution: Dissolve 15g of ammonium carbonate in 500m*l* of aqueous ammonia (2+25).

15.6.2 Flame Atomic Absorption Method

a) Target Element and quantitation range

Element	Percent Contained (%)
Cd	0.0001 – 1.0
Fe	0.0002 – 0.3
Pb	0.001 – 2.0
Pb	0.0001 – 0.02 (Iron hydroxide precipitation separation)

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current, slit width and flame conditions, refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions.

- Cd

Reagents:

1) Cd standard solution (20μg of Cd/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Zn solution: Decompose 100g of zinc (99.995wt%) in 400m*l* of hydrochloric acid, and heat until concentrated to a syrupy consistency. After cooling to ambient temperature, add 400m*l* to dissolve, and transfer to a 500m*l* volumetric flask. Add 20g of powdered zinc (for arsenic reagent analysis), and set aside for about 30 minutes, shaking occasionally to mix. After bringing up to volume with water, pass the solution through Type 5A dry filter paper. (200mg of Zn/m*l*)

Procedure:

1) The sample solution pretreated according to step a) can be measured directly, or use an aliquot (to which hydrochloric acid (1+1) is added so that the ratio of hydrochloric acid to sample solution is 10m*l* /100m*l*) for measurement.

   For blank measurement, perform the same pretreatment on the reagent as that for the sample, diluting only the standard solution. The value obtained from measuring this solution can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, transfer 25m*l* of Zn solution (200mg of Zn/m*l*) to each of several 100m*l* volumetric flasks. Add 20m*l* of hydrochloric acid (1+1) to each flask. Add Cd standard solution (20μg of Cd/m*l*) in increasing volumes from 0 – 30.0m*l* (containing 0 – 600μg of Cd), and bring the solutions up to volume with water. If an aliquot of the sample solution was used, the volume of zinc solution added should be adjusted so that it contains about the same amount of zinc in the sample aliquot.

Measurement:

Measurement wavelength: 228.8nm

Calibration curve range : 0.1 – 6μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 11)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Fe

Reagents:

1) Fe standard solution (20μg of Fe/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Zn solution: Same as for Cd, Reagent, 2)

Procedure:

1) Same as for Cd, Procedure, 1)
2) For the standard solutions to be used in generating the calibration

curve, transfer 25m*l* of Zn solution (200mg of Zn/m*l*) to each of several 100m*l* volumetric flasks. Add 20m*l* of hydrochloric acid (1+1) to each flask. Add Fe standard solution (20μg of Fe/m*l*) in increasing volumes from 0 – 40.0m*l* (containing 0 – 800μg of Fe), and bring the solutions up to volume with water. If an aliquot of the sample solution was used, the volume of zinc solution added should be adjusted so that it contains about the same amount of zinc in the sample aliquot.

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.5 – 8μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 17)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Pb

Reagents:

1) Pb standard solution (20μg of Pb/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Zn solution: Same as for Cd, Reagent, 2)

Procedure:

1) Same as for Cd, Procedure, 1)
2) For the standard solutions to be used in generating the calibration curve, transfer 25m*l* of Zn solution (200mg of Zn/m*l*) to each of several 100m*l* volumetric flasks. Add 20m*l* of hydrochloric acid (1+1) to each flask. Add Pb standard solution (20μg of Pb/m*l*) in increasing volumes from 0 – 50.0m*l* (containing 0 – 1000μg of Pb), and bring the solutions up to volume with water. If an aliquot of the sample solution was used, the volume of zinc solution added should be adjusted so that it contains about the same amount of zinc in the sample aliquot.

Measurement:

Measurement wavelength: 283.3nm

Calibration curve range : 1 – 10μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 27)

- Pb (Iron hydroxide precipitation separation)

  Reagents:

  1) Pb Standard solution (20μg of Pb/m*l*): Same as Pb, Reagent, 1)
  2) Ferric ammonium sulfate (III) solution: Same as described in Note for Sample Pretreatment, Pb (Iron hydroxide precipitation separation)

  Procedure:

  1) The sample solution pretreated according to step b) may be measured just as it is. For blank measurement, perform the same pretreatment on the reagent only as that performed on the sample. The value obtained from measuring this solution can be used to correct the value obtained in sample measurement.
  2) For the standard solutions used in generating the calibration curve, transfer 5m*l* of the ferric ammonium sulfate (III) solution to each of several 50m*l* volumetric flasks, and add 10m*l* of hydrochloric acid (1+1) to each flask. Add Pb standard solution (20μg of Pb/m*l*) in increasing volumes from 0 – 50.0m*l* (containing 0 – 500μg of Pb), and bring the solutions up to volume with water. Use these solutions for generating the calibration curve.

  Measurement:

  Measurement wavelength: 217.0nm

  Calibration curve range : 0.5 – 5μg /m*l*

  Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 26)

15.7 Nickel and Nickel Alloy Cast Metal Analysis Method

Reference materials: Japan Industrial Standard

Nickel / Nickel Alloy Cast Metal Lead Quantitation Method	JIS H 1272
Nickel / Nickel Alloy Cast Metal Iron Quantitation Method	JIS H 1273
Nickel / Nickel Alloy Cast Metal Manganese Quantitation Method	JIS H 1274
Nickel Alloy Cast Metal Chrome Quantitation Method	JIS H 1279

15.7.1 Sample Pretreatment

a) Cr

Weigh out 0.20g of sample and transfer to a 200m*l* beaker. Cover with a watch glass, add 10m*l* of hydrochloric acid and 3m*l* of nitric acid. Heat gently until decomposition is complete, and then continue heating until the nitrogen oxide is driven off. After cooling to ambient temperature, rinse the bottom of the watch glass with water and remove the watch glass. Then transfer the solution to a 100m*l* volumetric flask, and after adding 10m*l* of potassium disulfide (100 g/*l*), bring up to volume with water.

b) Cu, Fe, Mn

Weigh out 0.20g of sample and transfer to a 200m*l* beaker. Cover with a watch glass, add 20m*l* of nitric acid (1+1). Heat gently until decomposition is complete, and then continue heating until the nitrogen oxide is driven off. After cooling to ambient temperature, rinse the bottom of the watch glass with water and remove the watch glass. Then transfer the solution to a 100m*l* volumetric flask and bring up to volume with water.

15.7.2 Flame Atomic Absorption Method

a) Target Element and quantitation range

Element	Percent Contained (%)
Cr	0.05 – 2.0
Cu	0.05 – 2.0
Fe	0.05 – 2.0
Mn	0.05 – 2.0

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current, slit width and flame conditions, refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions.

- Cr

  Reagents:

  1) Cr standard solution (100μg of Cr/m*l*)
  2) Cr standard solution (500μg of Cr/m*l*)

     For 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards
  3) Metallic nickel: (>99.9wt%, containing <0.005wt% Cr, Cu, Fe and <0.001wt% of Mn)
  4) Metallic iron: (>99.9wt%, containing <0.005wt% Cr)
  5) Molybdenum solution (10μg of Mo/m*l*): Dissolve 18.4g of 7-molybdic acid-6-ammonium-4-hydrate in about 600m*l* of warm water. After cooling to ambient temperature, transfer to a 1000m*l* volumetric flask using water, and bring up to volume with water. (Prepare each time)

  Procedure:

  1) The sample solution pretreated according to step a) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cr standard solution. The value obtained can be used to correct the value obtained in sample measurement.
  2) For the standard solutions used in generating the calibration curve, weigh out 0.13g of metallic nickel and 0.010g of metallic iron, transfer these to several 200m*l* beakers, and perform the same operations as described in the pretreatment step a). Transfer each of these to a 100m*l* volumetric flask using water, and add 10m*l* of potassium disulfide solution (100g/*l*) and 6m*l* of molybdenum solution (10μg of Mo/m*l*). When the amount of Cr in the sample is 0.05 – 0.5%, add increasing volumes of Cr standard solution (100μg of Cr/m*l*) from 0 – 6.0m*l* (containing 0 – 600μg of Cr), or when the amount of Cr in the sample is 0. 5 – 2.0%, add increasing volumes of

Cr standard solution (500μg of Cr/m*l*) from 0 – 4.0m*l* (containing 0 – 2000μg of Cr), and bring up to volume with water.

Note: For nickel-lead alloy cast metal, add the same amount of nickel and lead as contained in the weighed sample.

Measurement:

Measurement wavelength: 357.9nm

Calibration curve range : 0.5 – 20μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 13)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Cu

Reagents:

1) Cu standard solution (100μg of Cu/m*l*)
2) Cu standard solution (500μg of Cu/m*l*)

   For 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards
3) Metallic nickel: Same as for Cr, Reagent, 3)

Procedure:

1) The sample solution pretreated according to step b) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cu standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions used in generating the calibration curve, weigh out 0.20g of metallic nickel, transfer to each of several 200m*l* beakers, and perform the same operations as described in the pretreatment step b). Transfer each of these to a 100m*l* volumetric flask using water. When the amount of Cu in the sample is 0.05 – 0.5%, add increasing volumes of Cu standard solution (100μg of Cu/m*l*) from 0 – 6.0m*l* (containing 0 – 600μg of Cu), or when the amount of Cu in the sample is 0. 5 – 2.0%, add increasing volumes of Cu standard solution (500μg of Cu/m*l*) from 0 – 4.0m*l* (containing 0

– 2000μg of Cu), and bring up to volume with water.

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.5 – 20μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Fe

Reagents:

1) Fe standard solution (100μg of Fe/m*l*)
2) Fe standard solution (500μg of Fe/m*l*)

   For 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards
3) Metallic nickel: Same as for Cr, Reagent, 3)

Procedure:

1) The sample solution pretreated according to step b) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Fe standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions used in generating the calibration curve, weigh out 0.20g of metallic nickel, transfer to each of several 200m*l* beakers, and perform the same operations as described in the pretreatment step b). Transfer each of these to a 100m*l* volumetric flask using water. When the amount of Fe in the sample is 0.05 – 0.5%, add increasing volumes of Fe standard solution (100μg of Fe/m*l*) from 0 – 6.0m*l* (containing 0 – 600μg of Fe). When the amount of Fe in the sample is 0. 5 – 2.0%, add increasing volumes of Fe standard solution (500μg of Fe/m*l*) from 0 – 4.0m*l* (containing 0 – 2000μg of Fe), and bring up to volume with water.

Note: For nickel-lead alloy cast metal, add the same amount of nickel and lead as contained in the weighed sample.

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.5 – 20μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 17)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Mn

Reagents:

1) Mn standard solution (100μg of Mn/m*l*)
2) Mn standard solution (500μg of Mn/m*l*)
   For 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards
3) Metallic nickel: Same as for Cr, Reagent, 3)

Procedure:

1) The sample solution pretreated according to step b) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Mn standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions used in generating the calibration curve, weigh out 0.20g of metallic nickel, transfer to each of several 200m*l* beakers, and perform the same operations as described in the pretreatment step b). Transfer each of these to a 100m*l* volumetric flask using water. When the amount of Mn in the sample is 0.05 – 0.3%, add increasing volumes of Mn standard solution (100μg of Mn/m*l*) from 0 – 3.0m*l* (containing 0 – 300μg of Mn). When the amount of Mn in the sample is 0.3 – 2.0%, add increasing volumes of Mn standard solution (500μg of Mn/m*l*) from 0 – 4.0m*l* (containing 0 – 200μg of Mn), and bring up to volume with water.

Note: For nickel-lead alloy cast metal, add the same amount of nickel and lead as contained in the weighed sample.

Measurement:

Measurement wavelength: 279.5nm

Calibration curve range : 0.2 – 20μg /m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 22)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

15.8 Analysis Method for Nickel Material for Electron Tubes

Reference materials: Japan Industrial Standard

Quantitation Method for Magnesium in Nickel Material for Electron Tubes	JIS H 1423
Quantitation Method for Lead in Nickel Material for Electron Tubes	JIS H 1424
Quantitation Method for Iron in Nickel Material for Electron Tubes	JIS H 1425
Quantitation Method for Manganese in Nickel Material for Electron Tubes	JIS H 1426
Quantitation Method for Cobalt in Nickel Material for Electron Tubes	JIS H 1431

15.8.1 Sample Pretreatment

a) Co, Cu, Fe, Mn

- Sample weights

Weigh out the sample according to the amount of target element, referring to the table below.

Element	Percent contained (%)	Weight (g)	Nitric Acid (1+1) Volume added (m*l*)
Co	0.01 – 0.05	1.00	20
	0.05 – 0.50	0.10	20
Cu	0.005 – 0.01	5.00	50
	0.01 – 0.10	0.50	20
	0.01 – 0.20	0.20	20
Fe	0.01 – 0.05	1.00	20
	0.05 – 0.20	0.20	20
Mn	0.005 – 0.01	5.00	50
	0.01 – 0.10	0.50	20
	0.10 – 0.40	0.10	20

- Preparing sample solutions

Weigh out the sample, transfer to a 300m*l* beaker, cover with a watch glass, and add nitric acid (1+1). After heating gently to decompose, boil to drive off the nitrogen oxide. After cooling, transfer to a 100m*l* volumetric flask and bring up to volume with water.

b) Mg

- Sample weights

Weigh out the sample according to the amount of target element, referring to the table below.

Percent contained (%)	Weight (g)	Nitric Acid (1+1) Volume added (m*l*)
0.002・0.01	1.00	20
0.01 ・0.05	0.20	20
0.05 ・0.2	0.10	20

- Preparing sample solutions

Weigh out the sample, transfer to a 300m*l* beaker, cover with a watch glass, and add nitric acid (1+1). After heating gently to decompose, boil to drive off the nitrogen oxide. After cooling, transfer to a 100m*l* volumetric flask, add 5m*l* of strontium solution (10mg of Sr/m*l*), and bring up to volume with water.

Note: Strontium solution (10mg of Sr/m*l*): Dissolve 30.4g of strontium chloride (6-hydrate) in water, transfer to a 1000m*l* volumetric flask, and bring up to volume with water.

15.8.2 Flame Atomic Absorption Method

a) Target element and quantitation range

Element	Percent contained (%)
Co	0.01 – 0.50
Cu	0.005 – 0.20
Fe	0.01 – 0.20
Mg	0.002 – 0.20
Mn	0.005 – 0.40

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current, slit width and flame conditions, refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions.

- Co

  Reagents:

  1) Co standard solution (100μg of Co/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Metallic nickel: >99.9 %, containing little Co, Cu, Fe, Mg, Mn

  Procedure:

  1) The sample solution pretreated according to step a) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Co standard solution. The value obtained can be used to correct the value obtained in sample measurement.
  2) For standard solutions used in generating a calibration curve, weigh out the same amount of metallic nickel as the sample weight used, and transfer to each of several 300m*l* beakers. After performing the same heating procedure as for the sample to decompose, add increasing volumes of Co standard solution (100μg of Co/m*l*) from 0 – 5.0m*l* (containing 0 – 500μg of Co). After cooling, transfer to 100m*l* volumetric flasks, and bring up to volume with water.

  Measurement:

  Measurement wavelength: 240.7nm

  Calibration curve range : 0.5 – 5μg/m*l*

  Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 12)

- Cu

  Reagents:

  1) Cu standard solution (100μg of Cu/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Metallic nickel: Same as for Co, Reagent, 2)

  Procedure:

  1) The sample solution pretreated according to step a) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cu standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For standard solutions used in generating a calibration curve, weigh out the same amount of metallic nickel as the sample weight used, and transfer to each of several 300m*l* beakers. After performing the same heating procedure as for the sample to decompose, add increasing volumes of Cu standard solution (100μg of Cu/m*l*) from 0 – 5.0m*l* (containing 0 – 500μg of Cu). After cooling, transfer to 100m*l* volumetric flasks, and bring up to volume with water.

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.2 – 5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Fe

Reagents:

1) Fe standard solution (100μg of Fe/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Metallic nickel: Same as for Co, Reagent, 2)

Procedure:

1) The sample solution pretreated according to step a) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Fe standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For standard solutions used in generating a calibration curve, weigh out the same amount of metallic nickel as the sample weight used, and transfer to each of several 300m*l* beakers. After performing the same heating procedure as for the sample to decompose, add increasing volumes of Fe standard solution (100μg of Fe/m*l*) from 0 – 5.0m*l* (containing 0 – 500μg of Fe. After cooling, transfer to 100m*l* volumetric flasks, and bring up to volume with water.

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.5 – 5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 17)

- Mg

Reagents:

1) Mg standard solution (20μg of Mg/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Metallic nickel: Same as for Co, Reagent, 2)
3) Sr solution (10mg of Sr/m*l*): Same as describe in NOTE for Sample Pretreatment, b)

Procedure:

1) The sample solution pretreated according to step b) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Mg standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For standard solutions used in generating a calibration curve, weigh out the same amount of metallic nickel as the sample weight used, and transfer to each of several 300m*l* beakers. After performing the same heating procedure as for the sample to decompose, add increasing volumes of Mg standard solution (20μg of Mg/m*l*) from 0 – 10.0m*l* (containing 0 – 200μg of Mg). After cooling, transfer to 100m*l* volumetric flasks. Add 5m*l* of Sr solution (10mg of Sr/m*l*) to each flask, and bring up to volume with water.

Measurement:

Measurement wavelength: 285.2nm

Calibration curve range : 0.1 – 2μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 21)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Mn

  Reagents:

  1) Mn standard solution (100μg of Mn/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Metallic nickel: Same as for Co, Reagent, 2)

  Procedure:

  1) The sample solution pretreated according to step a) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Mn standard solution. The value obtained can be used to correct the value obtained in sample measurement.
  2) For standard solutions used in generating a calibration curve, weigh out the same amount of metallic nickel as the sample weight used, and transfer to each of several 300m*l* beakers. After performing the same heating procedure as for the sample to decompose, add increasing volumes of Mn standard solution (100μg of Mn/m*l*) from 0 – 5.0m*l* (containing 0 – 500μg of Mn). After cooling, transfer to 100m*l* volumetric flasks, and bring up to volume with water.

  Measurement:

  Measurement wavelength: 279.5nm

  Calibration curve range : 0.2 – 5μg/m*l*

  Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 22)

  Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

15.9 Nickel Ore Analysis Method

Reference materials:

Japan Industrial Standard, Nickel Ore Analysis Method JIS H 1151

15.9.1 Sample Pretreatment

a) Co, Cu, Fe, Mn

Weigh out 5.0g of sample to the nearest 10mg and transfer to a 300m*l* beaker. Cover with a watch glass, add 40m*l* of nitric acid (1+1). Heat gently until decomposition is complete, and then boil until the nitrogen oxide is driven off. After cooling to ambient temperature, rinse the bottom of the watch glass with water and remove the watch glass. Transfer this solution to a 100m*l* volumetric flask using water, and bring up to volume with water.

b) Pb

Weigh out 5.0g of sample to the nearest 10mg and transfer to a 500m*l* beaker. Cover with a watch glass, add 40m*l* of nitric acid (1+1). Heat gently until decomposition is complete, and then boil until the nitrogen oxide is driven off. After cooling, rinse the bottom of the watch glass with water, remove the watch glass, and add water to a total volume of about 150m*l*.

Add 5m*l* of iron (III) solution, and while mixing this solution, little by little add 20m*l* of aqueous ammonia, then add 50m*l* more. Next, add about 15g of ammonium carbonate and dissolve. Heat and boil for about 5 minutes, and then let stand for 1 – 2 hours at 60 – 80°C. Filter out the precipitate using Type 5A filter paper, and rinse several times using warm ammonium rinse solution. Discard the rinse liquid and filtrate.

Using warm water, rinse the precipitate on the filter paper back into the original beaker. Place the beaker under the funnel, and drip 10m*l* of a nitric acid mixture onto the filter paper to dissolve any remaining precipitate on the filter paper and in the beaker. Then rinse the filter paper thoroughly with warm hydrochloric acid (1+50). Heat the solution until nearly evaporated. Add 5m*l* of hydrochloric acid (1+1) and 5m*l* of water, then heat to dissolve the salts. After cooling to ambient temperature, transfer to a 25m*l* volumetric flask, and bring up to volume with water.

Note:

1) Iron (III) solution: Dissolve 0.50g of iron (≥99.9%) in 20m*l* of

hydrochloric acid (1+1) and 2m*l* of hydrogen peroxide, heat to completely dissolve and continue heating to drive off hydrogen peroxide. After cooling to ambient temperature, dilute to 100m*l* using water (Fe 5μg/m*l*).

2) Ammonia rinse solution: Dissolve 15g of ammonium carbonate in 500m*l* of aqueous ammonia (2+50).

15.9.2 Flame Atomic Absorption Method

a) Target element and quantitation range

Element	Percent Contained (%)
Co	0.001 – 0.50
Cu	0.0005 – 0.50
Fe	0.0005 – 1.0
Mn	0.0001 – 0.004
Pb	0.0002 – 0.01

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current, slit width and flame conditions, refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions.

- Co

Reagents:

1) Co standard solution (50μg of Co/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Nickel: >99.9wt%, with known amount of Co, and amount of Co less than that in the sample.

Procedure:

1) When the sample solution pretreated according to step a) contains less than 0.01 % of Co, the solution is measured as it is. If the solution contains more than 0.01 % of Co, use an aliquot having a Co concentration which is within the calibration curve concentration range. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no

added Co standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For standard solutions used in generating a calibration curve, weigh out 5.0g of nickel, and transfer to each of several 300m*l* beakers. After performing the same procedure as for pretreatment of the sample using step a), transfer to 100m*l* volumetric flasks using water, add increasing volumes of Co standard solution (50μg of Co/m*l*) from 0 – 10.0m*l* (containing 0 – 500μg of Co), and bring up to volume with water.

   If an aliquot of the sample solution is used, weigh out 5.0g of nickel, and transfer to each of several 300m*l* beakers. After performing the same procedure as for pretreatment of the sample using step a), transfer to 100m*l* volumetric flasks using water, and bring up to volume with water. From this solution, take aliquots, each of which are equivalent in volume to the original aliquot taken from the sample solution, and transfer these to several 100m*l* volumetric flasks. Add increasing volumes of Co standard solution (50μg of Co/m*l*) from 0 – 10.0m*l* (containing 0 – 500μg of Co), and bring up to volume with water.

Measurement:

Measurement wavelength: 240.7nm

Calibration curve range : 0.5 – 5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 12)

- Cu

Reagents:

1) Cu standard solution (50μg of Cu/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Nickel: >99.9 %, with known amount of Cu, and amount of Cu less than that in the sample.

Procedure:

1) When the sample solution pretreated according to step a) contains less than 0.01 % of Cu, the solution is measured as it is. If the solution contains more than 0.01 % of Cu, use an aliquot having a Cu concentration which is within the calibration curve concentration range.

For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cu standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For standard solutions used in generating a calibration curve, weigh out 5.0g of nickel, and transfer to each of several 300m*l* beakers. After performing the same procedure as for pretreatment of the sample using step a), transfer to 100m*l* volumetric flasks using water, add increasing volumes of Cu standard solution (50μg of Cu/m*l*) from 0 – 10.0m*l* (containing 0 – 500μg of Cu), and bring up to volume with water.

   If an aliquot of the sample solution is used, weigh out 5.0g of nickel, and transfer to each of several 300m*l* beakers. After performing the same procedure as for pretreatment of the sample using step a), transfer to 100m*l* volumetric flasks using water, and bring up to volume with water. From this solution, take aliquots, each of which are equivalent in volume to the original aliquot taken from the sample solution, and transfer these to several 100m*l* volumetric flasks. Add increasing volumes of Cu standard solution (50μg of Cu/m*l*) from 0 – 10.0m*l* (containing 0 – 500μg of Cu), and bring up to volume with water.

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.2 – 5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 15)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Fe

Reagents:

1) Fe standard solution (50μg of Fe/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Nickel: >99.9wt%, with known amount of Fe, and amount of Fe less than that in the sample.

Procedure:

1) When the sample solution pretreated according to step a) contains less than 0.01 % of Fe, the solution is measured as it is. If the solution contains more than 0.01 % of Fe, use an aliquot having a Fe concentration which is within the calibration curve concentration range. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Fe standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For standard solutions used in generating a calibration curve, weigh out 5.0g of nickel, and transfer to each of several 300m*l* beakers. After performing the same procedure as for pretreatment of the sample using step a), transfer to 100m*l* volumetric flasks using water, add increasing volumes of Fe standard solution (50μg of Fe/m*l*) from 0 – 10.0m*l* (containing 0 – 500μg of Fe), and bring up to volume with water.

   If an aliquot of the sample solution is used, weigh out 5.0g of nickel, and transfer to each of several 300m*l* beakers. After performing the same procedure as for pretreatment of the sample using step a), transfer to 100m*l* volumetric flasks using water, and bring up to volume with water. From this solution, take aliquots, each of which are equivalent in volume to the original aliquot taken from the sample solution, and transfer these to several 100m*l* volumetric flasks. Add increasing volumes of Fe standard solution (50μg of Fe/m*l*) from 0 – 10.0m*l* (containing 0 – 500μg of Fe), and bring up to volume with water.

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.2 – 5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

- Mn

Reagents:

1) Mn standard solution (20μg of Mn/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

2) Nickel: >99.9wt%, with known amount of Mn, and amount of Mn less than that in the sample.

Procedure:

1) The sample solution pretreated according to step a) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Mn standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For standard solutions used in generating a calibration curve, weigh out 5.0g of nickel, and transfer to each of several 300m*l* beakers. After performing the same procedure as for pretreatment of the sample using step a), transfer to 100m*l* volumetric flasks using water, add increasing volumes of Mn standard solution (20μg of Mn/m*l*) from 0 – 10.0m*l* (containing 0 – 200μg of Mn), and bring up to volume with water.

Measurement:

Measurement wavelength: 279.5nm

Calibration curve range : 0.05 – 2μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 22)

- Pb

Reagents:

1) Pb standard solution (50μg of Pb/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Iron (III) solution: Prepared as described in the NOTE for Sample Pretreatment, b) Pb

Procedure:

1) The sample solution pretreated according to step b) is measured as it is. For blank measurement, perform the same pretreatment on the reagent containing no sample as that performed on the sample. This solution is measured, and the value obtained can be used to correct the value obtained in sample measurement.
2) For standard solutions used in generating a calibration curve, transfer increasing volumes of Pb standard solution (50μg of Pb/m*l*) from 0 –

10.0m*l* (containing 0 – 500μg of Pb) to several 25m*l* volumetric flasks. To each of these, add 5m*l* of iron (III) solution and 5m*l* of hydrochloric acid (1+1), and bring up to volume with water.

Measurement:

Measurement wavelength: 217.0nm

Calibration curve range : 0.4 – 20μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 26)

15.10 Lead Ore Analysis Method

Reference Materials:

Japan Industrial Standard, Lead Ore Analysis Method JIS H 1121

15.10.1 Sample Pretreatment

Weigh out 5.0g of sample to the nearest 10mg, and transfer to a 300m*l* beaker. Cover with at watch glass, add 10m*l* of tartaric acid solution (500 g/*l*) and 35m*l* of nitric acid (1+4). After heating gently to decompose, boil to drive off the nitrogen oxide. After cooling to ambient temperature, rinse the bottom surface of the watch glass and remove the watch glass. Transfer the solution to a 100m*l* volumetric flask using water, and bring up to volume with water.

15.10.2 Flame Atomic Absorption Method

a) Target element and quantitation range

Element	Percent Contained (%)
Ag	0.0002 – 0.004
Bi	0.001 – 0.15
Cu	0.0005 – 0.05
Fe	0.0005 – 0.05
Sb	0.002 – 0.15
Zn	0.0005 – 0.015

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current, slit width and flame conditions, refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions.

- Ag

Reagents:

1) Ag standard solution (20μg of Ag/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Lead: >99.99 %, containing no Ag, or if present, with known amount of Ag, and containing less than that in the sample.

Procedure:

1) The sample solution pretreated according to step a) is measured as it is. For blank measurement, when preparing the standards for generating

the calibration curve, prepare and measure a solution containing no added Ag standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For standard solutions used in generating a calibration curve, weigh out several 5.0g portions of lead, and after performing the same procedure as that in pretreatment of the sample, transfer these to 100m*l* volumetric flasks using water. Add increasing volumes of Ag standard solution (20μg of Ag/m*l*) from 0 – 10.0m*l* (containing 0 – 200μg of Ag), and bring up to volume with water.

Measurement:

Measurement wavelength: 328.1nm

Calibration curve range : 0.1 – 2μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 1)

- Bi

Reagents:

1) Bi standard solution (200μg of Bi/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Lead: >99.99 %, containing no Bi, or if present, with known amount of Bi, and containing less than that in the sample.

Procedure:

1) When the pretreated sample solution contains 0.001 – 0.06 % of Bi, it can be measured as it is. If it contains 0.06 – 0.15 %, take an exactly 20m*l* aliquot from the sample solution and transfer it to a 50m*l* volumetric flask. Add 3m*l* of tartaric acid solution (500 g/*l*) and 10m*l* of nitric acid (1+4), and bring up to volume with water.

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Bi standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For standard solutions used in generating the calibration curve, if the Bi content is 0.001 – 0.06 %, weigh out several 5.0g portions of lead, perform the same pretreatment as that used for the sample, and transfer these to several 100m*l* volumetric flasks using water. Add

increasing volumes of Bi standard solution (200μg of Bi/m*l*) from 0 – 15.0m*l* (containing 0 – 3000μg of Bi), bring up to volume with water, and use these solutions for measurement.

If the Bi content is 0.06 – 0.15 %, weigh out several 2.0g portions of lead, perform the same pretreatment as that used for the sample, and transfer these to several 100m*l* volumetric flasks using water. The rest of the procedure is the same as that used when the Bi content is 0.001 – 0.06 %.

Measurement:

Measurement wavelength: 223.1nm

Calibration curve range : 0.5 – 3μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 8)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Cu

Reagents:

1) Cu standard solution (100μg of Cu/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Lead: >99.99 %, containing no Cu, or if present, with known amount of Cu, and containing less than that in the sample.

Procedure:

1) When the pretreated sample solution contains 0.0005 – 0.02 % of Cu, it can be measured as it is. If it contains 0.02 – 0.05 %, accurately take a 20m*l* aliquot from the sample solution and transfer it to a 50m*l* volumetric flask. Add 3m*l* of tartaric acid solution (500 g/*l*) and 10m*l* of nitric acid (1+4), and bring up to volume with water.

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cu standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For standard solutions used in generating the calibration curve, if the Cu content is 0.0005 – 0.02 %, weigh out several 5.0g portions of

lead, perform the same pretreatment as that used for the sample, and transfer these to several 100m*l* volumetric flasks using water. Add increasing volumes of Cu standard solution (100μg of Cu/m*l*) from 0 – 10.0m*l* (containing 0 – 1000μg of Cu), bring up to volume with water, and use these solutions for measurement.

If the Cu content is 0.02 – 0.05 %, weigh out several 2.0g portions of lead, perform the same pretreatment as that used for the sample, and transfer these to several 100m*l* volumetric flasks using water. The rest of the procedure is the same as that used when the Cu content is 0.0005 – 0.02 %.

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.2 – 10μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 15)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Fe

Reagents:

1) Fe standard solution (100μg of Fe/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Lead: >99.99 %, containing no Fe, or if present, with known amount of Fe, and containing less than that in the sample.

Procedure:

1) When the pretreated sample solution contains 0.0005 – 0.02 % of Fe, it can be measured as it is. If it contains 0.02 – 0.05 %, accurately take a 20m*l* aliquot from the sample solution and transfer it to a 50m*l* volumetric flask. Add 3m*l* of tartaric acid solution (500 g/*l*) and 10m*l* of nitric acid (1+4), and bring up to volume with water.

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Fe standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For standard solutions used in generating the calibration curve, if the Fe content is 0.0005 – 0.02 %, weigh out several 5.0g portions of lead, perform the same pretreatment as that used for the sample, and transfer these to several 100m*l* volumetric flasks using water. Add increasing volumes of Fe standard solution (100μg of Fe/m*l*) from 0 – 10.0m*l* (containing 0 – 1000μg of Fe), bring up to volume with water, and use these solutions for measurement.

If the Fe content is 0.02 – 0.05 %, weigh out several 2.0g portions of lead, perform the same pretreatment as that used for the sample, and transfer these to several 100m*l* volumetric flasks using water. The rest of the procedure is the same as that used when the Fe content is 0.0005 – 0.02 %.

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.2 – 10μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

- Sb

Reagents:

1) Sb standard solution (200μg of Sb/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Lead: >99.99 %, containing no antimony, or if present, with known amount of antimony, and containing less than that in the sample.

Procedure:

1) When the pretreated sample solution contains 0.002 – 0.06 % of Sb, it can be measured as it is. If it contains 0.06 – 0.15 %, accurately take a 20m*l* aliquot from the sample solution and transfer it to a 50m*l* volumetric flask. Add 3m*l* of tartaric acid solution (500 g/*l*) and 10m*l* of nitric acid (1+4), and bring up to volume with water.

For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no

added Sb standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For standard solutions used in generating the calibration curve, if the Sb content is 0.002 – 0.06 %, weigh out several 5.0g portions of lead and perform the same pretreatment as that used for the sample. Transfer these to several 100m*l* volumetric flasks using water. Add increasing volumes of Sb standard solution (200μg of Sb/m*l*) from 0 – 15.0m*l* (containing 0 – 3000μg of Sb), bring up to volume with water, and use these solutions for measurement.

   If the Sb content is 0.06 – 0.15 %, weigh out several 2.0g portions of lead, perform the same pretreatment as that used for the sample, and transfer these to several 100m*l* volumetric flasks using water. The rest of the procedure is the same as that used when the Sb content is 0.002 – 0.06 %.

Measurement:

Measurement wavelength: 217.6nm

Calibration curve range : 1 – 30μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 32)

- Zn

Reagents:

1) Zn standard solution (20μg of Zn/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Lead: >99.99 %, containing no zinc, or if present, with known amount of zinc, and containing less than that in the sample.

Procedure:

1) When the pretreated sample solution contains 0.0005 – 0.006 % of Zn, it can be measured as it is. If it contains 0.006 – 0.015 %, take an exactly 20m*l* aliquot from the sample solution and transfer it to a 50m*l* volumetric flask. Add 3m*l* of tartaric acid solution (500 g/*l*) and 10m*l* of nitric acid (1+4), and bring up to volume with water.

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Zn standard solution. The value obtained can be used to correct

the value obtained in sample measurement.

2) For standard solutions used in generating the calibration curve, if the Zn content is 0.0005 – 0.006 %, weigh out several 5.0g portions of lead, perform the same pretreatment as that used for the sample, and transfer these to several 100m*l* volumetric flasks using water. Add increasing volumes of Zn standard solution (20μg of Zn/m*l*) from 0 – 15.0m*l* (containing 0 – 300μg of Zn), bring up to volume with water, and use these solutions for measurement.

   If the Zn content is 0.006 – 0.015 %, weigh out several 2.0g portions of lead, perform the same pretreatment as that used for the sample, and transfer these to several 100m*l* volumetric flasks using water. The rest of the procedure is the same as that used when the Zn content is 0.0005 – 0.006 %.

Measurement:

Measurement wavelength: 213.9nm

Calibration curve range : 0.2 – 3μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 44)

Note: If the standard solution absorbance exceeds 0.5, adjust the burner angle so that an absorbance of 0.5 is obtained for the standard solution with the highest concentration.

15.11 Tin Ore Analysis Method

Reference Materials:

Japan Industrial Standard, Tin Ore Analysis Method JIS H 1141

15.11.1 Sample Pretreatment

a) Direct method

Weigh out 1.00g of sample to the nearest 1mg and transfer to a 200m*l* beaker. Cover with a watch glass, little by little add 20m*l* of acid mixture (3 parts hydrochloric acid, 1 part nitric acid) to decompose. After the intense reaction subsides, heat gently until decomposition is complete. After cooling to ambient temperature, rinse the bottom surface of the watch glass and remove the watch glass. Transfer the solution to a 100m*l* volumetric flask using water, and bring up to volume with water.

b) Tin separation method

Weigh out 2.00g of sample to the nearest 1mg and transfer to a 200m*l* beaker. Cover with a watch glass, little by little add 10m*l* of acid mixture (3 parts hydrochloric acid, 1 part nitric acid) to decompose. After the intense reaction subsides, heat gently until decomposition is complete. After cooling, rinse the bottom surface of the watch glass and remove the watch glass. Add 10m*l* of sulfuric acid (1+1), and heat until volatilization of the white fumes of sulfuric acid occurs. After allowing to cool for several minutes, add 20m*l* of hydrobromic acid, and heat until thick white fumes are generated. After allowing to cool for several minutes, add 10m*l* of hydrobromic acid, and heat again until thick white fumes are generated. After cooling to ambient temperature, transfer the solution to a 25m*l* volumetric flask using water, and bring up to volume with water.

15.11.2 Electrical Heating Atomic Absorption Method

a) Target element and quantitation range

Element	Percent Contained (%)
As	0.0003 – 0.01 (Direct method)
Sb	0.0003 – 0.01 (Direct method)

Note: If the high purity tin mentioned in the next section cannot be obtained, this method is not applicable.

b) Measurement procedure

Measurement is conducted using the following procedure. For the lamp current and slit width, refer to Cookbook Section 4, Item 7.5 Element Specific Measurement Conditions.

- As

  Reagents:

  1) As standard solution (0.5μg of As/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
  2) Tin: High purity tin (>99.999 %), containing <0.00003 % arsenic

  Procedures:

  1) When the sample solution pretreated according to step a) contains less than 0.001 % of As, transfer a 50m*l* aliquot from the sample solution to a 100m*l* volumetric flask and bring up to volume with water. If the sample solution contains more than 0.001 %, transfer a 5.0m*l* aliquot from the sample solution to a 100m*l* volumetric flask and bring up to volume with water.

     When preparing the standards for generating the calibration curve, prepare and measure prepare and measure a solution containing no added As standard solution for blank measurement. The value obtained can be used to correct the value obtained in sample measurement.
  2) For the standard solutions to be used for generating the calibration curve, when the amount of As in the sample is less than 0.001 %, weigh out several 1.00g portions of tin and transfer these to separate 200m*l* beakers. For each of these, perform the pretreatment procedure a) just as that for the sample, transfer to a 100m*l* volumetric flask using water, and bring up to volume with water. Transfer exactly 50m*l* aliquots from each of these solutions to several 100m*l* volumetric flasks. Add increasing volumes of the As standard solution (0.5μg of As/m*l*) from 0 – 10.0m*l* (As content from 0 – 5μg), and bring up to volume with water.

     When the amount of As in the sample is more than 0.001 %, weigh out several 1.00g portions of tin and transfer these to separate 200m*l*

beakers. For each of these, perform the pretreatment procedure a) just as that for the sample, transfer to a 100m*l* volumetric flask using water, and bring up to volume with water. Transfer exactly 5.0m*l* aliquots from each of these solutions to several 100m*l* volumetric flasks. Add increasing volumes of the As standard solution (0.5μg of As/m*l*) from 0 – 10.0m*l* (As content from 0 – 5μg), and bring up to volume with water.

Measurement:

Measurement wavelength: 193.7nm

Calibration curve range : 2 – 50 ng/m*l*

Tube : High density graphite tube

Sample injection volume : 20μ*l*

Heating conditions :

	TEMP (Ž)	TIME (sec)	HEAT	GAS (*l*/min)
STAGE 1	120	20	R	0.2
2	250	10	R	0.2
3	400	10	R	1.0
4	400	20	S	1.0
5	400	3	S	0.0
6	2200	3	S	0.0
7	2500	2	S	1.0

- Sb

Reagents:

1) Sb standard solution (0.5μg of Sb/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards.
2) Tin: High purity tin (>99.999 %), containing <0.00003 % antimony

Procedures:

1) When the sample solution pretreated according to step a) contains less than 0.001 % of Sb, transfer a 50m*l* aliquot from the sample solution to a 100m*l* volumetric flask and bring up to volume with water. If the sample solution contains more than 0.001 %, transfer a

5.0m*l* aliquot from the sample solution to a 100m*l* volumetric flask and bring up to volume with water.

For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Sb standard solution. The value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, when the amount of Sb in the sample is less than 0.001 %, weigh out several 1.00g portions of tin and transfer these to separate 200m*l* beakers. For each of these, perform the pretreatment procedure a) just as that for the sample, transfer to a 100m*l* volumetric flask using water, and bring up to volume with water. Transfer exactly 50m*l* aliquots from each of these solutions to several 100m*l* volumetric flasks. Add increasing volumes of the Sb standard solution (0.5μg of Sb/m*l*) from 0 – 10.0m*l* (Sb content from 0 – 5μg), and bring up to volume with water.

   When the amount of Sb in the sample is more than 0.001 %, weigh out several 1.00g portions of tin and transfer these to separate 200m*l* beakers. For each of these, perform the pretreatment procedure a) just as that for the sample, transfer to a 100m*l* volumetric flask using water, and bring up to volume with water. Transfer exactly 5.0m*l* aliquots from each of these solutions to several 100m*l* volumetric flasks. Add increasing volumes of the Sb standard solution (0.5μg of Sb/m*l*) from 0 – 10.0m*l* (Sb content from 0 – 5μg), and bring up to volume with water.

Measurement:

Measurement wavelength: 217.6nm
Calibration curve range : 5 – 50 ng/m*l*
Tube : Pyrolized graphite tube
Sample injection volume : 20μ*l*

Heating conditions :

	TEMP (Ž)	TIME (sec)	HEAT	GAS (*l*/min)
STAGE 1	120	20	R	0.2
2	250	10	R	0.2
3	400	10	R	1.0
4	400	20	S	1.0
5	400	3	S	0.0
6	2200	3	S	0.0
7	2500	2	S	1.0

15.11.3 Flame Atomic Absorption Method

a) Target elements and quantitation range

Element	Percent contained (%)	
Cu	0.001 – 0.05	(Direct method)
	0.0001 – 0.005	(Tin separation method)
Fe	0.001 – 0.05	(Direct method)
	0.0003 – 0.005	(Tin separation method)
Pb	0.002 – 0.10	(Direct method)
	0.0002 – 0.01	(Tin separation method)

Note: If the high purity tin mentioned in the next section cannot be obtained, this method is not applicable.

b) Measurement procedure

Measurement is conducted using the following procedure. Refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions, for the lamp current, slit width and flame conditions.

- Cu I (Direct method)

  Reagents:

  1) Cu standard solution (20μg of Cu/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Tin: >99.99 %, with known amount of Cu, and containing less than that in the sample.

Procedure:

1) The sample solution pretreated according to step a) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cu standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, weigh out several 1.00g portions of tin and transfer to separate 200m*l* beakers. After performing the same pretreatment procedure as that for the sample, described in step a), transfer to several 100m*l* volumetric flasks using water, and add increasing volumes of the Cu standard solution (20μg of Cu/m*l*) from 0 – 25.0m*l* (Cu content from 0 – 500μg), and bring up to volume with water.

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.1 – 5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 15)

- Cu II (Tin separation method)

Reagents:

1) Cu standard solution (10μg of Cu/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to step b) is measured as it is. For blank measurement, reagent containing no sample is pretreated using the same procedure as that used for the sample. After measurement, the value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, add increasing volumes of the Cu standard solution (10μg of Cu/m*l*) from 0 – 10.0m*l* (Cu content from 0 – 100μg) to several 25m*l* volumetric flasks, add 10m*l* of nitric acid (1+2) and bring up to volume with water.

Measurement: Same as for Cu I

- Fe I (Direct method)

  Reagents:

  1) Fe standard solution (20μg of Fe/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Tin: >99.99 %, with known amount of Fe, and containing less than that in the sample.

  Procedure:

  1) The sample solution pretreated according to step a) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Fe standard solution. The value obtained can be used to correct the value obtained in sample measurement.
  2) For the standard solutions to be used in generating the calibration curve, weigh out several 1.00g portions of tin and transfer to separate 200m*l* beakers. After performing the same pretreatment procedure as that for the sample, described in step a), transfer to several 100m*l* volumetric flasks using water, and add increasing volumes of the Fe standard solution (20μg of Fe/m*l*) from 0 – 25.0m*l* (Fe content from 0 – 500μg), and bring up to volume with water.

  Measurement:

  Measurement wavelength: 248.3nm

  Calibration curve range : 0.1 – 5μg/m*l*

  Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

- Fe II (Tin separation method)

  Reagents:

  Fe standard solution (10μg of Fe/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

  Procedure:

  1) The sample solution pretreated according to step b) is measured as it is. Pre-treat reagent containing no sample using the same procedure as that used for the sample for blank measurement. After measurement, the value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, add increasing volumes of the Fe standard solution (10μg of Fe/m*l*) from 0 – 10.0m*l* (Fe content from 0 – 100μg) to several 25m*l* volumetric flasks, add 10m*l* of nitric acid (1+2) and bring up to volume with water.

Measurement: Same as for Fe I

- Pb I (Direct method)

  Reagents:

  1) Pb standard solution (100μg of Pb/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Tin: >99.99 %, with known amount of Pb, and containing less than that in the sample.

  Procedure:

  1) The sample solution pretreated according to step a) is measured as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Pb standard solution. The value obtained can be used to correct the value obtained in sample measurement.
  2) For the standard solutions to be used in generating the calibration curve, weigh out several 1.00g portions of tin and transfer to separate 200m*l* beakers. After performing the same pretreatment procedure as that for the sample, described in step a), transfer to several 100m*l* volumetric flasks using water, and add increasing volumes of the Pb standard solution (100μg of Pb/m*l*) from 0 – 10.0m*l* (Pb content from 0 – 1000μg), and bring up to volume with water.

  Measurement:

  Measurement wavelength: 217.0nm

  Calibration curve range : 0.2 – 10μg/m*l*

  Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 26)

- Pb II (Tin separation method)

  Reagents:

  Pb standard solution (20μg of Pb/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to step b) is measured as it is. For blank measurement, reagent containing no sample is pretreated using the same procedure as that used for the sample. After measurement, the value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, add increasing volumes of the Pb standard solution (20μg of Pb/m*l*) from 0 – 10.0m*l* (Pb content from 0 – 200μg) to several 25m*l* volumetric flasks, add 10m*l* of nitric acid (1+2) and bring up to volume with water.

Measurement: Same as for Pb I

15.12 Cadmium Ore Analysis Method

Reference Materials:

Japan Industrial Standard, Cadmium Ore Analysis Method JIS H 1161

15.12.1 Sample Pretreatment

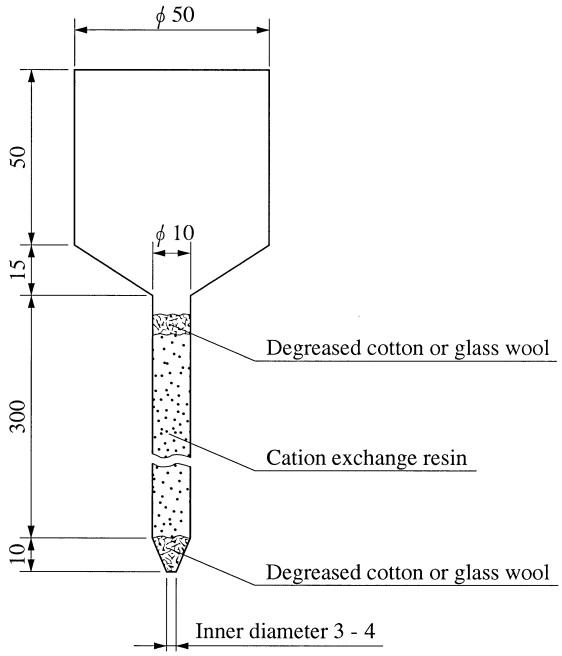
a) Direct method

Weigh out 5.0g of sample and transfer to a 300m*l* beaker. Cover with a watch glass, and add 50m*l* of nitric acid (1+3). After the intense reaction subsides, rinse the bottom surface of the watch glass and remove the watch glass. Transfer this solution to a 100m*l* volumetric flask and bring up to volume with water.

b) Ion exchange separation method

Weigh out 5.0g of sample and transfer to a 300m*l* beaker. Cover with a watch glass, add 40m*l* of acid mixture (10 parts hydrochloric acid, 2 parts nitric acid, 10 parts water). After the intense reaction subsides, heat gently until decomposition is complete. Continue heating until the solution is concentrated to a syrupy consistency. After cooling, rinse the bottom surface of the watch glass and remove the watch glass. Add 20m*l* of hydrochloric acid (1+1), heat until the salts are dissolved. Then cool to room temperature.

Prepare a cation exchange column as displayed in the following figure, and loosely pack in the bottom of the glass tube a 5 – 10mm thick layer of degreased cotton or glass wool. Pour onto this bed about 18m*l* of a water-swollen slurry of strongly acidic cation exchange resin (particle diameter 74 – 149μm, exchange capacity greater than 1.9 m equivalence/m*l*). After the resin settles, loosely pack on top of this a 5mm thick layer of degreased cotton or glass wool. Adjust the degreased cotton or glass wool so that the flow rate through the column is 1 – 2m*l*/min. Then pass through the column 50m*l* of sodium hydroxide solution (10g/*l*) and 50m*l* of water, in that order, and store the resin in NaR form.(R= an organic group)



Unit: mm

Cation Exchange Column Example

Pass the solution, which has been cooled to room temperature, through the prepared cation exchange column. Next, rinse the interior wall of the beaker twice with 10m*l* of hydrochloric acid (1+16). After passing each of these rinsees through the column, once again pass through 80m*l* of hydrochloric acid (1+16) to elute the cadmium. This elute may be discarded. Place the column over a 200m*l* receiving beaker and continue passing through the column 10m*l* of hydrochloric acid (1+3) to elute the lead. Heat the elute for about 10 minutes, then cool to ambient temperature, transfer to a 25m*l* volumetric flask, and bring up to volume with water.

Note:

1) Depending on the column used, the volume of hydrochloric acid (1+16) necessary to elute the cadmium may vary, so be sure to verify the elution curve, and if necessary, adjust the amount of acid used.
2) Depending on the column used, the volume of hydrochloric acid (1+3) necessary to elute the lead may vary, so be sure to verify the elution curve, and if necessary, adjust the amount of acid used.

15.12.2 Flame Atomic Absorption Method

a) Target element and quantitation range

Element	Percent Contained (%)	
Cu	0.0002 – 0.05	(Direct method)
	0.0001 – 0.005	(Ion exchange separation method)
Fe	0.0002 – 0.02	(Direct method)
	0.0001 – 0.002	(Ion exchange separation method)
Pb	0.0005 – 0.05	(Direct method)
	0.0001 – 0.01	(Ion exchange separation method)
Zn	0.0001 – 0.01	(Direct method)
	0.0001 – 0.001	(Ion exchange separation method)

Note: In the Direct method, if the high purity cadmium cannot be obtained, this method is not applicable.

b) Measurement procedure

Measurement is conducted using the following procedure. Refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions, for the lamp current, slit width and flame conditions.

- Cu I (Direct method)

Reagents:

1) Cu standard solution (20μg of Cu/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Cadmium: >99.99 %, with known amount of Cu, and containing less than that in the sample.

Procedure:

1) When the sample solution pretreated according to step a) contains less than 500μg of Cu, the solution is measured as it is. When there is more than 500μg of Cu, transfer a 10.0m*l* aliquot from the sample solution to a 50m*l* volumetric flask. Add 5m*l* of nitric acid (1+1), and bring up to volume with water.
2) For the standard solutions to be used in generating the calibration curve, when there is less than 500μg of Cu, weigh out several 5.0g portions of cadmium and transfer to separate 300m*l* beakers. After

performing the same pretreatment procedure as that for the sample, described in step a), transfer to several 100m*l* volumetric flasks using water. Add increasing volumes of the Cu standard solution (20μg of Cu/m*l*) from 0 – 25.0m*l* (Cu content from 0 – 500μg), and bring up to volume with water.

When there is more than 500μg of Cu, weigh out several 5.0g portions of cadmium and transfer to separate 300m*l* beakers. After performing the same pretreatment procedure as that for the sample, described in step a), transfer to several 100m*l* volumetric flasks using water, and bring up to volume with water. Transfer several 10.0m*l* aliquots from this solution to separate 50m*l* volumetric flasks. Add 5m*l* of nitric acid (1+1) and then add increasing volumes of the Cu standard solution (20μg of Cu/m*l*) from 0 – 12.5m*l* (Cu content from 0 – 250μg), and bring up to volume with water.

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.1 – 5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 15)

- Cu II (Ion exchange separation method)

  Reagents:

  Cu standard solution (10μg of Cu/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

  Procedure:

  1) When the sample solution pretreated according to step b) contains less than 100μg of Cu, the solution is measured as it is. When there is more than 100μg of Cu, transfer a 10.0m*l* aliquot from the sample solution to a 25m*l* volumetric flask, and bring up to volume with water.

     For blank measurement, pre-treat reagent containing no sample using the same procedure as that used for the sample. After measurement, the value obtained can be used to correct the value obtained in sample measurement.

  2) For the standard solutions to be used in generating the calibration curve, add increasing volumes of the Cu standard solution (10μg of

Cu/m*l*) from 0 – 10.0m*l* (Cu content from 0 – 100μg) to several 25m*l* volumetric flasks. Add 10m*l* of hydrochloric acid (1+1) and bring up to volume with water.

Measurement: Same as for Cu I

- Fe I (Direct method)

  Reagents:

  1) Fe standard solution (20μg of Fe/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Cadmium: >99.99 %, with known amount of Fe, and containing less than that in the sample.

  Procedure:

  1) When the sample solution pretreated according to step a) contains less than 500mg of Fe, the solution is measured as it is. When there is more than 500mg of Fe, transfer a 10.0m*l* aliquot from the sample solution to a 50m*l* volumetric flask. Add 5m*l* of nitric acid (1+1), and bring up to volume with water.
  2) For the standard solutions to be used in generating the calibration curve, when there is less than 500μg of Fe, weigh out several 5.0g portions of cadmium and transfer to separate 300m*l* beakers. After performing the same pretreatment procedure as that for the sample, described in step a), transfer to several 100m*l* volumetric flasks using water, add increasing volumes of the Fe standard solution (20μg of Fe/m*l*) from 0 – 25.0m*l* (Fe content from 0 – 500μg), and bring up to volume with water.

     When there is more than 500μg of Fe, weigh out several 5.0g portions of cadmium and transfer to separate 300m*l* beakers. After performing the same pretreatment procedure as that for the sample, described in step a), transfer to several 100m*l* volumetric flasks using water, and bring up to volume with water. Transfer several 10.0m*l* aliquots from this solution to separate 50m*l* volumetric flasks. Add 5m*l* of nitric acid (1+1) and then add increasing volumes of the Fe standard solution (20μg of Fe/m*l*) from 0 – 10m*l* (Fe content from 0 – 200μg), and bring up to volume with water.

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.1 – 5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

- Fe II (Ion exchange separation method)

Reagents:

Fe standard solution (10μg of Fe/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to step b is measured as it is. For blank measurement, reagent containing no sample is pretreated using the same procedure as that used for the sample. After measurement, the value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, add increasing volumes of the Fe standard solution (10μg of Fe/m*l*) from 0 – 10.0m*l* (Fe content from 0 – 100μg) to several 25m*l* volumetric flasks. Add 10m*l* of hydrochloric acid (1+1) and bring up to volume with water.

Measurement: Same as for Fe I

- Pb I (Direct method)

Reagents:

1) Pb standard solution (50μg of Pb/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Cadmium: >99.99 %, with known amount of Pb, and containing less than that in the sample.

Procedure:

1) When the sample solution pretreated according to step a) contains less than 1000μg of Pb, the solution is measured as it is. When there is more than 1000μg of Pb, transfer a 10.0m*l* aliquot from the sample solution to a 50m*l* volumetric flask. Add 5m*l* of nitric acid (1+1), and bring up to volume with water.
2) For the standard solutions to be used in generating the calibration curve, when there is less than 1000μg of Pb, weigh out several 5.0g

portions of cadmium and transfer to separate 300m*l* beakers. After performing the same pretreatment procedure as that for the sample, described in step a), transfer to several 100m*l* volumetric flasks using water, add increasing volumes of the Pb standard solution (50μg of Pb/m*l*) from 0 – 20.0m*l* (Pb content from 0 – 1000μg), and bring up to volume with water.

When there is more than 1000μg of Pb, weigh out several 5.0g portions of cadmium and transfer to separate 300m*l* beakers. After performing the same pretreatment procedure as for the sample, described in step a), transfer to several 100m*l* volumetric flasks, and bring up to volume with water. Transfer several 10.0m*l* aliquots from this solution to separate 50m*l* volumetric flasks. Add 5m*l* of nitric acid (1+1) and then add increasing volumes of the Pb standard solution (50μg of Pb/m*l*) from 0 – 10.0m*l* (Pb content from 0 – 500μg), and bring up to volume with water.

Measurement:

Measurement wavelength: 217.0nm

Calibration curve range: 0.25 – 10μg/m*l*

Measurement conditions: Refer to Cookbook Section 3, Item 6.4, 26)

- Pb II (Ion exchange separation method)

  Reagents:

  Pb standard solution (25μg of Pb/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

  Procedure:

  1) When the sample solution pretreated according to step b) contains less than 250μg of Pb, the solution is measured as it is. When there is more than 250μg of Pb, transfer a 10.0m*l* aliquot from the sample solution to a 25m*l* volumetric flask, and bring up to volume with water.

     For blank measurement, reagent containing no sample is pretreated using the same procedure as that used for the sample. After measurement, the value obtained can be used to correct the value obtained in sample measurement.

  2) For the standard solutions to be used in generating the calibration curve, add increasing volumes of the Pb standard solution (25μg of

Pb/m*l*) from 0 – 10.0m*l* (Pb content from 0 – 250μg) to several 25m*l* volumetric flasks. Add 10m*l* of hydrochloric acid (1+1) and bring up to volume with water.

Measurement: Same as for Pb I

- Zn I (Direct method)

  Reagents:

  1) Zn standard solution (5μg of Zn/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Cadmium: >99.99 %, with known amount of Zn, and containing less than that in the sample.

  Procedure:

  1) When the sample solution pretreated according to step a) contains less than 100μg of Zn, the solution is measured as it is. When there is more than 100μg of Zn, transfer a 10.0m*l* aliquot from the sample solution to a 50m*l* volumetric flask. Add 5m*l* of nitric acid (1+1), and bring up to volume with water.
  2) For the standard solutions to be used in generating the calibration curve, when there is less than 100μg of Zn, weigh out several 5.0g portions of cadmium and transfer to separate 300m*l* beakers. After performing the same pretreatment procedure as that for the sample, described in step a), transfer to several 100m*l* volumetric flasks using water, add increasing volumes of the Zn standard solution (5μg of Zn/m*l*) from 0 – 20.0m*l* (Zn content from 0 – 100μg), and bring up to volume with water.

     When there is more than 100mg of Zn, weigh out several 5.0g portions of cadmium and transfer to separate 300m*l* beakers. After performing the same pretreatment procedure as that for the sample, described in step a), transfer to several 100m*l* volumetric flasks, and bring up to volume with water. Transfer several 10.0m*l* aliquots from this solution to separate 50m*l* volumetric flasks. Add 5m*l* of nitric acid (1+1) and then add increasing volumes of the Zn standard solution (5μg of Zn/m*l*) from 0 – 10.0m*l* (Zn content from 0 – 50μg), and bring up to volume with water.

Measurement:

Measurement wavelength: 213.9nm

Calibration curve range : 0.05 – 1μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 44)

- Zn II (Ion exchange separation method)

Reagents:

Zn standard solution (2μg of Zn/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) When the sample solution pretreated according to step b) contains less than 20μg of Zn, the solution is measured as it is. When there is more than 20μg of Zn, transfer a 10.0m*l* aliquot from the sample solution to a 25m*l* volumetric flask, and bring up to volume with water.

   For blank measurement, reagent containing no sample is pretreated using the same procedure as that used for the sample. After measurement, the value obtained can be used to correct the value obtained in sample measurement.

2) For the standard solutions to be used in generating the calibration curve, add increasing volumes of the Zn standard solution (2μg of Zn/m*l*) from 0 – 10.0m*l* (Zn content from 0 – 20μg) to several 25m*l* volumetric flasks. Add 10m*l* of hydrochloric acid (1+1) and bring up to volume with water.

Measurement: Same as for Zn I

15.13 Silver Ore Analysis Method

Reference Materials:

Japan Industrial Standard, Silver Ore Analysis Method JIS H1181

15.13.1 Sample Pretreatment

a) Bi, Pb

Weigh out 10g of sample, and transfer to a 300m*l* beaker. Cover with a watch glass, add 40m*l* of nitric acid, and heat gently to decompose. Add water to a volume of about 100m*l*. Add 5m*l* of ferric ammonium sulfate, and while mixing, add aqueous

ammonia. After the formation of iron hydroxide precipitate, add 20m*l* more of the aqueous ammonia. Next, add 5g of ammonium carbonate and heat. After boiling gently for about 10 minutes, set aside in a warm location (60 – 80°C) for 30 minutes. Separate the precipitate using Type 5A filter paper, and, after rinseing several times with warm ammonia rinse solution, transfer it to the original beaker by rinseing with warm water. Place the beaker under the filter, and drip 10m*l* of hydrochloric acid (1+1) over the filter paper to dissolve the precipitate on the filter paper and in the beaker. Rinse the filter paper thoroughly with warm water, and combine this liquid with that in the original beaker. Heat this solution to concentrate down to a volume of about 10m*l*.

Transfer the solution to a 100m*l* beaker using warm hydrochloric acid (1+50), add 2m*l* of sulfuric acid (1+1) and heat to concentrate until nearly evaporated. After cooling, add 5m*l* of hydrochloric acid (1+2), cover with a watch glass, heat to dissolve, and keep heating until nearly evaporated. After cooling once again, add 5m*l* of hydrochloric acid (1+2), cover with a watch glass, and heat to dissolve. After cooling, transfer to a 25m*l* volumetric flask using hydrochloric acid (1+50), and dilute up to volume.

Note:

1) Ferric ammonium sulfate: Dissolve 5g of ferric ammonium sulfate in nitric acid (1+100) and bring up to 100m*l*.
2) Ammonia rinse solution: Dissolve 5g of ammonium carbonate in 500m*l* of aqueous ammonia (1+19)

b) Cu, Fe

Weigh out 10g of sample, and transfer to a 500m*l* beaker. Cover with a

watch glass, add 40m*l* of sulfuric acid (1+1), and heat gently to decompose. Add warm water to a volume of about 100m*l*. While mixing this solution, little by little add 20m*l* of hydrochloric acid (1+1) to precipitate out the silver chloride, and keep mixing for 10 minutes longer.

After cooling, pass the solution through Type 5B filter paper, and rinse several times with hydrochloric acid (1+100). To the filtrate and rinse solution, add 2m*l* of sulfuric acid (1+1), and heat until nearly evaporated. After cooling, add 5m*l* of hydrochloric acid (1+2), heat to dissolve, and evaporate to near dryness. After cooling, once again add 5m*l* of hydrochloric acid (1+2), cover with a watch glass, and heat to dissolve. After cooling, transfer to a 25m*l* volumetric flask and bring up to volume with water.

15.13.2 Flame Atomic Absorption Method

a) Target element and quantitation range

Element	Percent Contained (%)
Bi	>0.0001
Cu	>0.0001
Fe	>0.0002
Pb	>0.0001

b) Measurement procedure

Measurement is conducted using the following procedure. Refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions, for the lamp current, slit width and flame conditions.

- Bi

Reagents:

1) Bi standard solution (10μg of Bi/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Ferric ammonium sulfate solution: Refer to NOTE 1) for Sample Pretreatment for a) Bi, Cu

Procedure:

1) The sample solution pretreated according to step a) is measured just as it is. When the percent content of Bi in the sample is high, use an

aliquot from the 25m*l* sample solution which contains 50 – 250μg of Bi, and use the same concentration of hydrochloric acid as the concentration of the sample.

At this time, for a blank test, take an appropriate amount of reagent containing no sample, perform the same pretreatment as that used for the sample, and then measure this solution. The obtained value may be used for the correction of the value obtained in sample measurement.

2) For the standard solutions used in generating the calibration curve, transfer increasing volumes of Bi standard solution (10μg of Bi/m*l*) from 0 – 25.0m*l* (containing 0 – 250μg of Bi) to several 100m*l* beakers. Add 5m*l* of ferric ammonium sulfate solution and 2m*l* of sulfuric acid (1+1), and heat until evaporated. Then follow the procedure described in the Sample Pretreatment for a) Bi, Cu after " from the addition of 5m*l* of the hydrochloric acid (1+2)" .

Measurement:

Measurement wavelength: 223.1nm

Calibration curve range : 1 – 10μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 8)

- Cu

Reagents:

Cu standard solution (10μg of Cu/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to step b) is measured just as it is. When the percent content of Cu in the sample is high, use an aliquot from the 25m*l* of sample solution which contains 25 – 100μg of Cu, and use the same concentration of chlorine as the concentration of the sample.

For a blank test, take an appropriate amount of reagent containing no sample, perform the same pretreatment as that used for the sample, and then measure this solution. The obtained value may be used for the correction of the value obtained in sample measurement.

2) For the standard solutions used in generating the calibration curve,

transfer increasing volumes of Cu standard solution (10μg of Cu/m*l*) from 0 – 10.0m*l* (containing 0 – 100μg of Cu) to several 25m*l* volumetric flasks. Add 6m*l* of hydrochloric acid (1+2) and bring up to volume with water.

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.2 – 4μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 15)

- Fe

Reagents:

Fe standard solution (10μg of Fe/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to step b) is measured just as it is. When the percent content of Fe in the sample is high, use an aliquot from the 25m*l* of sample solution which contains 25 – 100μg of Fe, and use the same concentration of hydrochloric acid as the concentration of the sample.

   At this time, for a blank test, take an appropriate amount of reagent containing no sample, and perform the same pretreatment as that used for the sample. Then measure this solution. The obtained value may be used for the correction of the value obtained in sample measurement.

2) For the standard solutions used in generating the calibration curve, transfer increasing volumes of Fe standard solution (10μg of Fe/m*l*) from 0 – 10.0m*l* (containing 0 – 100μg of Fe) to several 25m*l* volumetric flasks. Add 6m*l* of hydrochloric acid (1+2) and bring up to volume with water.

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.5 – 4μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

- Pb

  Reagents:

  1) Pb standard solution (10μg of Pb/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Ferric ammonium sulfate solution: Refer to NOTE 1) for Sample Pretreatment for a) Bi, Cu

  Procedure:

  1) The sample solution pretreated according to step a) is measured just as it is. When the percent content of Pb in the sample is high, use an aliquot from the 25m*l* sample solution which contains 50 – 250μg of Pb, and use the same concentration of hydrochloric acid as the concentration of the sample.

     At this time, for a blank test, take an appropriate amount of reagent containing no sample. Perform the same pretreatment as that used for the sample, and then measure this solution. The obtained value may be used for the correction of the value obtained in sample measurement.
  2) For the standard solutions used in generating the calibration curve, transfer increasing volumes of Pb standard solution (10μg of Pb/m*l*) from 0 – 25.0m*l* (containing 0 – 250μg of Pb) to several 100m*l* beakers. Add 5m*l* of ferric ammonium sulfate solution and 2m*l* of sulfuric acid (1+1), and heat until evaporated. Then follow the Sample Pretreatment procedure described above after " from the addition 5m*l* of the hydrochloric acid (1+2)....." . Perform measurement using the resulting solution.

  Measurement:

  Measurement wavelength: 283.3nm

  Calibration curve range : 1 – 10μg/m*l*

  Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 27)

15.14 Magnesium Ore Analysis Method

Reference Materials:

Japan Industrial Standard, Magnesium Ore Analysis Method JIS H 1321

15.14.1 Sample Pretreatment

Weigh out 1.0g of sample and transfer to a 300m*l* beaker. Add about 10m*l* of water, cover with watch glass, and little by little add 20m*l* of hydrochloric acid (1+1). When the reaction has subsided, add 1m*l* of hydrogen peroxide. Heat until decomposition is complete, and then cool. Transfer to a 100m*l* volumetric flask and bring up to volume with water.

15.14.2 Flame Atomic Absorption Method

a) Target elements and quantitation range

Element	Percent Contained (%)
Cu	>0.0002 (DADDC - APDC extraction method)
Fe	>0.001 (DADDC - APDC extraction method)
Mn	0.0005 – 0.02
Ni	>0.0002 (DADDC - APDC extraction method)
Zn	0.001 – 0.05

b) Measurement procedure

Measurement is conducted using the following procedure. Refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions, for the lamp current, slit width and flame conditions.

- Cu

Reagents:

1) Cu standard solution (5μg of Cu/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Tartaric acid solution (40 w/v%)
3) Diethylammonium diethyldithiocarbamate (DADDC) solution: Dissolve 0.5g of diethylammonium diethyldithiocarbamate in water, and dilute to 100m*l*. If there are impurities, filter and use the filtrate. (Prepare each time)
4) Ammonium pyrrolizidinedithiocarbamate (APDC) solution: Dissolve 0.5g of ammonium pyrrolizidinedithiocarbamate in water, and dilute to 100m*l*. If there are impurities, filter and use the filtrate.

5) n-butyl acetic acid

6) Methyl red solution: Dissolve 0.2g of methyl red in 90m*l* of ethanol, and dilute to 100m*l*.

Procedure:

1) Take an aliquot from the sample solution containing 5 – 20μg of Cu, and transfer to a 200m*l* beaker. Add 20m*l* of tartaric acid (40 w/v%), and dilute to about 80m*l* with water. Next, adjust to a pH of 5.8±0.2 using aqueous ammonia or hydrochloric acid solution. Transfer to a 200m*l* separating funnel, add water to bring volume to about 100m*l*, add 5m*l* of DADDC solution and 5m*l* of APDC solution and shake to mix. Add exactly 20m*l* of n-butyl acetic acid, shake vigorously for 3 minutes. After the contents separate into 2 layers, dehydrate the organic phase and use for measurement.

   For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure prepare and measure a solution containing no added Cu standard solution. The value obtained can be used to correct the value obtained in sample measurement.

   Note: For the dehydration procedure, pack dry filter paper (type 2) or degreased cotton into the bottom stem of the separating funnel. Remove the water by passing the organic phase through this, or transfer the organic phase to a stoppered Erlenmeyer flask, containing sodium sulfate (anhydrous), and shake to dehydrate.

2) For the standard solutions used in generating the calibration curve, accurately transfer increasing volumes of Cu standard solution (5μg of Cu/m*l*) from 0 – 4.0m*l* (containing 0 – 20μg of Cu) into several beakers. The remainder of the procedure is the same as described in 1). The resulting solution is used for measurement.

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.1 – 1μg/m*l*

Measurement Conditions :

Slit width : 0.5nm

Lamp mode : BGC-D_2

Burner height : 7mm

Support gas : Air

Fuel gas flow rate : C_2H_2 0.8*l*/min (If flame appears reddish when sample is sprayed, decrease the amount of sample suctioned.)

- Fe

Reagents:

1) Fe standard solution (5μg of Fe/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
2) Tartaric acid solution (40 w/v%)
3) Diethylammonium diethyldithiocarbamate (DADDC) solution
4) Ammonium pyrrolizidinedithiocarbamate (APDC) solution
5) n-butyl acetic acid
6) Methyl red solution

2) – 6) are prepared as described for Cu, Reagents, 2) – 6)

Procedure:

1) Take an aliquot from the sample solution containing 10 – 40μg of Fe, and transfer to a 200m*l* beaker. The remainder of the procedure is as described for Cu, Procedure, 1).
2) For the standard solutions used in generating the calibration curve, accurately transfer increasing volumes of Fe standard solution (10μg of Fe/m*l*) from 0 – 4.0m*l* (containing 0 – 40μg of Fe) into several beakers. The remainder of the procedure is the same as described in 1). The resulting solution is used for measurement.

Measurement:

Measurement wavelength : 248.3nm

Calibration curve range : 0.2 – 2μg/m*l*

Measurement Conditions :

Lamp current : 12 mA

Slit width : 0.2nm

Lamp mode : BGC-D_2

Burner height : 7mm

Support gas : Air

Fuel gas flow rate : C_2H_2 0.8*l*/min (If flame appears reddish when sample is sprayed, decrease the amount of sample suctioned.)

- Mn

  Reagents:

  1) Mn standard solution (20μg of Mn/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Magnesium chloride solution: Dissolve 105g of magnesium chloride in water, and dilute to 250m*l*.(20m*l* of this solution contains 1.0g of Mg).

  Procedure:

  1) The pretreated sample solution is measured just as it is.

     For blank measurement, take an appropriate amount of reagent containing no sample, and perform the same pretreatment as that used for the sample. Then measure this solution. The obtained value may be used for the correction of the value obtained in sample measurement.
  2) For the standard solutions used in generating the calibration curve, add 20m*l* of the magnesium chloride solution to each of several 100m*l* volumetric flask. Accurately add increasing volumes of Mn standard solution (20μg of Mn/m*l*) from 0 – 10m*l* (containing 0 – 200μg of Mn) into several beakers. Bring up to volume with water and use this solution for measurement.

  Measurement:

  Measurement wavelength : 279.5nm

  Calibration curve range : 0.2 – 2μg/m*l*

  Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 22)

- Ni

  Reagents:

  1) Ni standard solution (5μg of Ni/m*l*): Refer to Cookbook Section 2, Item 3 Preparing Standards
  2) Tartaric acid solution (40 w/v%)

3) Diethylammonium diethyldithiocarbamate (DADDC) solution
4) Ammonium pyrrolizidinedithiocarbamate (APDC) solution
5) n-butyl acetic acid
6) Methyl red solution

2) – 6) are prepared as described for Cu, Reagents, 2) – 6)

Procedure:

1) Take an aliquot from the sample solution containing 5 – 20μg of Ni, and transfer to a 200m*l* beaker. The remainder of the procedure is as described for Cu, Procedure, 1).
2) For the standard solutions used in generating the calibration curve, accurately transfer increasing volumes of Ni standard solution (5μg of Ni/m*l*) from 0 – 4.0m*l* (containing 0 – 20μg of Ni) into several beakers. The remainder of the procedure is the same as described in 1). The resulting solution is used for measurement.

Measurement:

Measurement wavelength: 232.0nm

Calibration curve range : 0.2 – 1.0μg/m*l*

Measurement Conditions :

Lamp current : 12 mA
Slit width : 0.2nm
Lamp mode : BGC-D_2
Burner height : 7mm
Support gas : Air
Fuel gas flow rate: C_2H_2 0.8*l*/min (If flame appears reddish when sample is sprayed, decrease the amount of sample suctioned.)

- Zn

Reagents:

1) Zn standard solution (5μg of Zn/m*l*)
2) Zn standard solution (2μg of Zn/m*l*)

For 1) and 2), refer to Cookbook Section 2, Item 3 Preparing Standards

3) Magnesium chloride solution: Same as described for Mn, Reagents, 2)

Procedure:

1) If the percent of Zn contained in the pretreated sample solution is less than 0.001 %, that solution is measured as it is. If the sample solution contains more than 0.001 % Zn, use an aliquot specified in the following table, and bring up to volume with water.

Percent Zn contained (%)	Aliquot (m*l*)	Volumetric flask (m*l*)
0.001 – 0.02	20	100
0.02 – 0.05	10	100

For blank measurement, perform the same procedure on the reagent containing no sample as that performed for pretreatment of the sample. Then measure this solution. The obtained value may be used for the correction of the value obtained in sample measurement.

2) For the standard solutions used to generate the calibration curve, if the amount of Zn in the sample solution is less than 0.001 %, transfer 20m*l* of magnesium chloride solution to each of several 100m*l* volumetric flasks. Accurately add increasing volumes of Zn standard solution (2μg of Zn/m*l*) from 0 – 5.0m*l* (containing 0 – 10μg of Zn), and bring up to volume with water.

If the amount of Zn is less than 0.001 %, transfer 4m*l* of hydrochloric acid (1+1) to each of several 100m*l* volumetric flasks. Accurately add increasing volumes of Zn standard solution (5μg of Zn/m*l*) from 0 – 10.0m*l* (containing 0 – 50μg of Zn), and bring up to volume with water.

Measurement:

Measurement wavelength: 213.9nm

Calibration curve range : 0.02 – 0.5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 44)

15.15 Solder Analysis Method

Reference Materials: Japan Industrial Standard, Solder Analysis Method JIS Z 3910

The solder referred to here corresponds to the following items specified in the Japan Industrial Standard JIS Z 3910:

Pb97.5Ag, Pb97.5SnAg1.5

Pb98Sn, Pb95Sn, Pb90Sn, Pb80Sn, Pb70Sn, Pb65Sn

Pb60Sn, Pb55Sn

Sn50Pb, Sn55Pb, Sn60Pb, Sn63Pb, Sn65Pb

Sn62PbAg2

Sn43PbBi14

Bi58Sn

15.15.1 Sample Pretreatment

a) Al

Weigh out 30g of sample and transfer to a 300m*l* beaker. Add 30m*l* of hydrobromic acid-bromine mixture and heat to decompose. To this add 20m*l* of sulfuric acid and heat until the white fumes of sulfuric acid are generated. Then, little by little add 10m*l* of hydrobromic acid. Heat to volatilize the tin, antimony, arsenic, etc. until the volume is concentrated to about 10m*l*. After cooling, add 50m*l* of water, and heat to dissolve the soluble salts.

After cooling, filter out the lead sulfate precipitate using Type 5C filter paper, and rinse with sulfuric acid (2+100). Transfer the filtrate and rinse liquid to a separate tall 300m*l* beaker using water, and heat until evaporated. Add 10m*l* of hydrochloric acid and 10m*l* of nitric acid to this dry beaker, and heat to dissolve all the soluble salts, concentrating the liquid to a volume of about 20m*l*. After cooling to ambient temperature, transfer to 50m*l* volumetric flask using water, and bring up to volume with water.

Note: Hydrobromic acid-bromine mixture: Add 20m*l* of bromine to 180m*l* of hydrogen bromide, and mix by shaking.

b) Bi, Cd, Cu, Fe, Pb, Sb, Zn

Weigh out 1.0g of sample. Transfer to a 300m*l* beaker, cover with a watch glass, add 50m*l* of acid mixture, and after heating to decompose, continue heating until total volume decreases to 10 – 20m*l*. After cooling to ambient temperature, transfer to a 100m*l* volumetric flask and bring up to volume with water. Let stand for about one hour, and then filter using Type 5C filter paper.

Note: Acid mixture: 85 parts hydrochloric acid, 10 parts nitric acid, 5 parts water

15.15.2 Flame Atomic Absorption Method

a) Target elements and quantitation range

Element	Percent contained (%)
Al	<0.01
Bi	<0.05
Cd	<0.005
Cu	<0.05
Fe	<0.05
Pb	<0.1
Sb	<0.5
Zn	<0.005

b) Measurement procedure

Measurement is conducted using the following procedure. Refer to Cookbook Section 3, Item 6.4 Element Specific Measurement Conditions, for the lamp current, slit width and flame conditions.

- Al

Reagents:

Al standard solution (50μg of Al/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to step a) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Al standard solution is prepared and measured, and the value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, weigh out several 3.0g lots containing the elements contained in the sample at about the same ratio as that in the sample. Transfer

these to several tall 300m*l* beakers, respectively. Add increasing volumes of the Al standard solution (50μg of Al/m*l*) from 0 – 10.0m*l* (Al content from 0 – 500μg). The remainder of the procedure is the same as that used in the sample pretreatment step a) for Al. Use the resulting solution for measurement.

Measurement:

Measurement wavelength: 309.3nm

Calibration curve range : 2 – 10μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 2)

- Bi (Not applicable for Sn43PbBi14)

Reagents:

Bi standard solution (100μg of Bi/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to step b) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Bi standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, weigh out several 1.0g lots containing the elements contained in the sample at about the same ratio as that in the sample. Transfer these to several 300m*l* beakers, respectively. Add increasing volumes of the Bi standard solution (100μg of Bi/m*l*) from 0 – 10.0m*l* (Bi content from 0 – 1000μg). The remainder of the procedure is the same as that used in the sample pretreatment step b). Use the resulting solution for measurement.

Measurement:

Measurement wavelength: 223.1nm

Calibration curve range : 1 – 10μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 8)

- Cd

  Reagents:

  Cd standard solution (20μg of Cd/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards

  Procedure:

  1) The sample solution pretreated according to step b) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cd standard solution. The value obtained can be used to correct the value obtained in sample measurement.
  2) For the standard solutions to be used in generating the calibration curve, weigh out several 1.0g lots containing the elements contained in the sample at about the same ratio as that in the sample, and transfer these to several 300m*l* beakers, respectively. Add increasing volumes of the Cd standard solution (20μg of Cd/m*l*) from 0 – 5.0m*l* (Cd content from 0 – 100μg). The remainder of the procedure is the same as that used in the sample pretreatment step b). Use the resulting solution for measurement.

  Measurement:

  Measurement wavelength: 228.8nm

  Calibration curve range : 0.05 – 1μg/m*l*

  Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 11)

- Cu

  Reagents:

  Cu standard solution (50μg of Cu/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards

  Procedure:

  1) The sample solution pretreated according to step b) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Cu standard solution. The value obtained can be used to correct the value obtained in sample measurement.
  2) For the standard solutions to be used in generating the calibration

curve, weigh out several 1.0g lots containing the elements contained in the sample at about the same ratio as that in the sample, and transfer these to several 300m*l* beakers, respectively. Add increasing volumes of the Cu standard solution (50μg of Cu/m*l*) from 0 – 10.0m*l* (Cu content from 0 – 500μg). The remainder of the procedure is the same as that used in the sample pretreatment step b). Use the resulting solution for measurement.

Measurement:

Measurement wavelength: 324.8nm

Calibration curve range : 0.2 – 5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 15)

- Fe

Reagents:

Fe standard solution (50μg of Fe/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to step b) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Fe standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, weigh out several 1.0g lots containing the elements contained in the sample at about the same ratio as that in the sample, and transfer these to several 300m*l* beakers, respectively. Add increasing volumes of the Fe standard solution (50μg of Fe/m*l*) from 0 – 10.0m*l* (Fe content from 0 – 500μg). The remainder of the procedure is the same as that used in the sample pretreatment step b). Use the resulting solution for measurement.

Measurement:

Measurement wavelength: 248.3nm

Calibration curve range : 0.2 – 5μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 16)

- Pb (Applicable for Bi58Sn, Sn96.5Ag and Sn95Sb)

  Reagents:

  Pb standard solution (100μg of Pb/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards

  Procedure:

  1) The sample solution pretreated according to step b) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Pb standard solution. The value obtained can be used to correct the value obtained in sample measurement.
  2) For the standard solutions to be used in generating the calibration curve, weigh out several 1.0g lots containing the elements contained in the sample at about the same ratio as that in the sample, and transfer these to several 300m*l* beakers, respectively. Add increasing volumes of the Pb standard solution (100μg of Pb/m*l*) from 0 – 20.0m*l* (Pb content from 0 – 2000μg). The remainder of the procedure is the same as that used in the sample pretreatment step b). Use the resulting solution for measurement.

  Measurement:

  Measurement wavelength: 283.3nm

  Calibration curve range : 2 – 20μg/m*l*

  Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 27)

- Sb (Not applicable for Sn95Sb)

  Reagents:

  Sb standard solution (500μg of Sb/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards

  Procedure:

  1) The sample solution pretreated according to step b) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Sb standard solution. The value obtained can be used to correct the value obtained in sample measurement.
  2) For the standard solutions to be used in generating the calibration

curve, weigh out several 1.0g lots containing the elements contained in the sample at about the same ratio as that in the sample, and transfer these to several 300m*l* beakers, respectively. Add increasing volumes of the Sb standard solution (500μg of Sb/m*l*) from 0 – 10.0m*l* (Sb content from 0 – 5000μg). The remainder of the procedure is the same as that used in the sample pretreatment step b). Use the resulting solution for measurement.

Measurement:

Measurement wavelength: 217.6nm

Calibration curve range : 2 – 50μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 32)

- Zn

Reagents:

Zn standard solution (20μg of Zn/m*l*): refer to Cookbook Section 2, Item 3 Preparing Standards

Procedure:

1) The sample solution pretreated according to step b) is measured just as it is. For blank measurement, when preparing the standards for generating the calibration curve, prepare and measure a solution containing no added Zn standard solution. The value obtained can be used to correct the value obtained in sample measurement.
2) For the standard solutions to be used in generating the calibration curve, weigh out several 1.0g lots containing the elements contained in the sample at about the same ratio as that in the sample, and transfer these to several 300m*l* beakers, respectively. Add increasing volumes of the Zn standard solution (20μg of Sb/m*l*) from 0 – 5.0m*l* (Zn content from 0 – 100μg). The remainder of the procedure is the same as that used in the sample pretreatment step b). Use the resulting solution for measurement.

Measurement:

Measurement wavelength: 213.9nm

Calibration curve range : 0.5 – 1μg/m*l*

Measurement conditions : Refer to Cookbook Section 3, Item 6.4, 44)