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**Methods for chemical analysis of
aluminium dross for iron and steel
making**

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Foreword

This Japanese Industrial Standard has been revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Aluminium Association (JAA)/Japanese Standards Association (JSA) with a draft being attached, based on the provision of Article 12, paragraph (1) of the Industrial Standardization Act applied mutatis mutandis pursuant to the provision of Article 16 of the said Act. This edition replaces the previous edition (**JIS G 2404 : 2015**), which has been technically revised.

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Methods for chemical analysis of aluminium dross for iron and steel making

1 Scope

This Japanese Industrial Standard specifies the methods for determination of metallic aluminium, nitrogen, chlorine, fluorine, total silicon, aluminium oxide and carbon, and the moisture content in aluminium dross which is used as a flux in the manufacture of iron and steel.

WARNING Persons performing tests in accordance with this Standard should exercise adequate caution in handling high pressure gas in the ICP atomic emission spectrometry and the infrared absorption after combustion, as well as in the use and disposal of dangerous chemicals (e.g. bromine, hydrofluoric acid, organic solvent), and should make efforts to prevent hazards and protect the environment.

2 Normative references

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

- JIS G 2402 *Aluminium dross for iron and steel making*
- JIS G 2403 *Aluminium dross for iron and steel making — Sampling and sample preparation procedures*
- JIS H 4160 *Aluminium and aluminium alloy foils*
- JIS H 6201 *Platinum crucibles for chemical analysis*
- JIS K 0050 *General rules for chemical analysis*
- JIS K 0101 *Testing methods for industrial water*
- JIS K 0102 *Testing methods for industrial wastewater*
- JIS K 0115 *General rules for molecular absorptiometric analysis*
- JIS K 0116 *General rules for atomic emission spectrometry*
- JIS K 0127 *General rules for ion chromatography*
- JIS K 0211 *Technical terms for analytical chemistry (General part)*
- JIS K 0215 *Technical terms for analytical chemistry (Analytical instrument part)*
- JIS K 0970 *Piston pipettes*
- JIS K 8005 *Reference materials for volumetric analysis*
- JIS R 1306 *Porcelain combustion boats for chemical analysis*

JIS Z 2615 *General rules for determination of carbon in metallic materials*

JIS Z 8401 *Rounding of numbers*

JIS Z 8710 *Temperature measurement — General requirement*

3 Terms and definitions

For the purpose of this Standard, the following terms and definitions, and those given in **JIS G 2402**, **JIS K 0127**, **JIS K 0211** and **JIS K 0215** apply.

3.1

thermal hydrolysis

operation and method of decomposing a sample in a reaction tube by heating it while passing steam and gas through the tube

3.2

carrier gas

gas used in thermal hydrolysis to introduce the components generated in the decomposition in the steam flow into the absorbing solution

4 General requirements

General requirements of chemical analysis shall be in accordance with **JIS K 0050** and the following.

- a) The sampling and the sample preparation shall be in accordance with **JIS G 2403**.
- b) Water used for analyses shall be in accordance with Classification A2 or A3 specified in Annex D of **JIS K 0050**.
- c) Reagents and gases used for analyses, if relevant **JIS**s exist, shall be of the highest specified grade or appropriate for the intended analytical purpose. For reagents and gases for which no **JIS** exists, those having a quality not interfering with the analysis shall be used.
- d) General requirements of ICP atomic emission spectrometry shall be in accordance with **JIS K 0116**.
- e) General requirements of ion chromatography shall be in accordance with **JIS K 0127**.
- f) General requirements of molecular absorptiometric analysis shall be in accordance with **JIS K 0115**.
- g) General requirements of carbon determination shall be in accordance with **JIS Z 2615**.
- h) The analytical values shall be expressed by the percentage (mass fraction) to the mass of sample weighed out and rounded by Rule A of **JIS Z 8401** to the number of digits specified for each analytical method in this Standard.

5 Determination of metallic aluminium

5.1 Classification of determination methods

The determination of metallic aluminium shall be performed by one of the following methods.

- a) **ICP atomic emission spectrometry for decomposed and separated metallic aluminium** This method is applicable to samples with metallic aluminium content of not less than 1 % (mass fraction).
- b) **Zinc back-titration for decomposed and separated metallic aluminium** This method is applicable to samples with metallic aluminium content of not less than 5 % (mass fraction).
- c) **Gas volumetric method by dissolution with hydrochloric acid** This method is used for the daily control analysis of metallic aluminium, and applicable to samples with metallic aluminium content of 5 % (mass fraction) or over to and excluding 80 % (mass fraction).
- d) **Measurement using reaction solution temperature** This method is used for the daily control analysis of metallic aluminium, and applicable to samples with metallic aluminium content of 20 % (mass fraction) or over to and excluding 60 % (mass fraction). This method is influenced by impurities such as nitrogen compound of aluminium and magnesium.

NOTE Methods **a)** and **b)** are used as reference methods, and methods **c)** and **d)** are used for the purpose of daily quality control and for the acceptance testing performed upon delivery of products to the customer.

5.2 ICP atomic emission spectrometry for decomposed and separated metallic aluminium

5.2.1 Summary

The sample is decomposed with bromine in methanol and metallic aluminium is dissolved. After the undissolved residue is filtered, the solution is made a definite volume and aluminium is determined by ICP atomic emission spectrometry.

5.2.2 Reagents

The reagents shall be as follows.

- a) **Hydrochloric acid (1+1)**
- b) **Hydrogen peroxide**
- c) **Bromine**
- d) **Argon**
- e) **Methanol**
- f) **Yttrium solution (Y : 1 mg/ml)** Weigh out 1.270 g of yttrium (III) oxide [not less than 99.9 % (mass fraction)] and transfer to a conical beaker (200 ml). Cover the beaker with a watch glass, add 50 ml of hydrochloric acid (1+1), and heat to de-

compose. Cool to the ordinary temperature, and remove the watch glass after washing its under-surface with water. Put the washings into the conical beaker. Transfer this solution into a 1 000 ml volumetric flask using water and dilute to the mark with water.

Alternatively, a commercially available solution suitable for the intended analytical purpose may be used.

- g) **Aluminium standard solution (Al : 1 mg/ml)** Weigh out 1.000 g of aluminium [not less than 99.9 % (mass fraction)] and transfer to a conical beaker (500 ml). Cover the beaker with a watch glass, add 20 ml of hydrochloric acid (1+1) and 20 ml of nitric acid (1+1), and gently heat to decompose. When the decomposition is complete, gently boil for 2 min to 3 min to expel nitrogen oxides. Cool in running water to the ordinary temperature, and remove the watch glass after washing its under-surface with water. Put the washings into the conical beaker. Transfer the solution into a 1 000 ml volumetric flask using water and dilute to the mark with water.

Alternatively, a commercially available solution suitable for the intended analytical purpose may be used.

NOTE Some commercial standard solutions are guaranteed with metrological traceability [e.g. standard solutions with certificates bearing a JCSS (Japan Calibration Service System) mark supplied in accordance with National Measurement Standards Provision System].

5.2.3 Apparatus

An ICP atomic emission spectrophotometer with the configuration specified in **JIS K 0116** shall be used.

5.2.4 Chemical analysis sample

The chemical analysis sample shall be in accordance with **JIS G 2403**.

NOTE The sample preparation according to **7.4 b)** of **JIS G 2403** is as follows : Pulverize the sample and pass through a sieve of 1.0 mm or 0.5 mm nominal aperture. Further pulverize the sample remaining on the sieve, and without passing the pulverized sample through the sieve, mix it well with the sample having passed the sieve, and reduce this mixture.

5.2.5 Sample mass

The mass of sample to be weighed out shall be 2.0 g, accurate to 1 mg.

5.2.6 Operation

5.2.6.1 Preparation of sample solution

The preparation of sample solution shall be as follows.

- Weigh out a sample and transfer to a dry conical beaker (500 ml).
- Add 200 ml of methanol [**5.2.2 e)**], insert a solid-stem thermometer (0 °C to 100 °C) into the beaker and cover the beaker with a dry watch glass. Add 10 ml of bromine [**5.2.2 c)**] using a dry pipette 1 ml by 1 ml at first, and when the reaction becomes

gentle, 2 ml by 2 ml. Maintain the solution temperature during this reaction at 40 °C or lower ¹⁾ by, if necessary, cooling the solution in running water or using other appropriate procedure.

Note ¹⁾ When the solution temperature exceeds 40 °C during decomposition, the aluminium compounds other than metallic aluminium start to be dissolved.

- c) After the addition of bromine is complete and the decomposition of metallic aluminium, etc. is almost complete, leave the solution to stand for about 1 h to allow a thorough decomposition.
- d) Remove the watch glass after washing its under-surface with water. Put the washings into the conical beaker. Filter the solution through a filter paper (Class 5C) or a membrane filter made of polytetrafluoroethylene or polycarbonate resin with a pore size of 1 µm, and wash the filter paper (or membrane filter) and the undissolved residue with a small amount of methanol four or five times. Receive both the filtrate and the washings in a 500 ml volumetric flask containing 40 ml of hydrochloric acid (1+1) [5.2.2 a)].
- e) Dilute to about 450 ml with water, cool in running water to the ordinary temperature, and dilute further to the mark with water.
- f) Take a 50 ml aliquot of this solution in a conical beaker (300 ml), add 5 ml of hydrochloric acid (1+1), and heat on a hot plate to allow to evaporate and concentrate to a volume of about 5 ml.
- g) Remove from the hot plate and allow to cool. Add 1 ml of hydrogen peroxide [5.2.2 b)], heat to remove bromine, and then further heat to concentrate to a syrupy consistency.
- h) Add 10 ml of hydrochloric acid (1+1) to dissolve the precipitated salts, and boil gently for 1 min to 2 min. Cool in running water to the ordinary temperature, transfer to a 100 ml volumetric flask using water and dilute to the mark with water.
- i) Take a 10 ml aliquot of this solution in a 100 ml volumetric flask, add 5 ml of yttrium solution [5.2.2 f)] and dilute to the mark with water.

5.2.6.2 Measurement

The measurement shall be as follows.

- a) Light the argon plasma of the ICP atomic emission spectrophotometer and set up the apparatus in a condition ready for stable measurement.
- b) Spray a part of the solution prepared according to 5.2.6.1 i) into the argon plasma of the ICP atomic emission spectrophotometer and measure the emission intensity at a wavelength of aluminium emission line of 309.271 nm, 309.284 nm, 396.152 nm or the like, and then at a wavelength of yttrium emission line of 371.03 nm or the like. If the spectrophotometer is capable of simultaneous background measurement, measure the background at a wavelength near the selected measuring wavelength at which aluminium and other coexisting elements do not interfere,

and subtract the measured background to determine the emission intensity of aluminium.

5.2.7 Blank test

The blank test shall not be carried out.

5.2.8 Preparation of calibration curve

The preparation of calibration curve shall be as follows.

- a) Add eight volumes of aluminium standard solutions [5.2.2 g)] respectively containing 0 mg, 0.5 mg, 1 mg, 3 mg, 5 mg, 10 mg, 15 mg and 20 mg of aluminium into eight separate 100 ml volumetric flasks. To each of these solutions, add 1 ml of hydrochloric acid (1+1), then 5 ml of yttrium solution [5.2.2 f)], and dilute to the mark with water.
- b) Perform the procedure in 5.2.6.2 using each of these solutions to measure the emission intensity of aluminium, and prepare a calibration curve by plotting the relation between the emission intensity and the amount of aluminium. In the case of using the intensity ratio method, determine the ratio of the emission intensity of aluminium to that of yttrium, and prepare a calibration curve by plotting the relation between this ratio and the amount of aluminium.

5.2.9 Calculation

Determine the amount of aluminium on the calibration curve prepared according to 5.2.8, and calculate the metallic aluminium content of the sample according to Formula (1) and round the result off to two decimal places :

$$Al = \frac{a \times \frac{500}{50} \times \frac{100}{10}}{W} \times 100 \dots\dots\dots (1)$$

where, *Al* : metallic aluminium content of sample [% (mass fraction)]
 a : amount of aluminium obtained from calibration curve (g)
 W: sample mass (g)

5.3 Zinc back-titration method for decomposed and separated metallic aluminium

5.3.1 Summary

The sample is decomposed with bromine in methanol and metallic aluminium is dissolved. The undissolved residue is filtered. Disodium dihydrogen ethylenediaminetetraacetate (EDTA2Na) is excessively added to the filtrate, and its excess is titrated with zinc by using a xylenol orange indicator. EDTA2Na combined with aluminium is isolated by adding sodium fluoride, and titrated similarly to the first titration by using zinc.

5.3.2 Reagents

The reagents shall be as follows.

- a) Hydrochloric acid (1+1)

- b) **Ammonia solution (1+2)**
- c) **Hydrogen peroxide**
- d) **Bromine**
- e) **Sodium fluoride saturated solution**, prepared by dissolving sodium fluoride in water in a polyethylene beaker. Store this solution in a polyethylene bottle.
- f) **Methanol**
- g) **Disodium dihydrogen ethylenediaminetetraacetate (EDTA2Na solution)** Weigh out 37.2 g of disodium dihydrogen ethylenediaminetetraacetate dihydrate, add 1 L of water to dissolve, and store in an airtight container made of polyethylene or other resin material.
- h) **0.1 mol/L zinc titrant** Weigh out 6.538 g of zinc [not less than 99.99 % (mass fraction)] to the nearest 1 mg, and transfer to a conical beaker (1 L). Add 100 ml of hydrochloric acid (1+1) [5.3.2 a)], heat to thoroughly decompose, and cool in running water to room temperature. Make this solution about 500 ml, add ammonia solution to make alkaline, then adjust to pH 5 to pH 6 by using acetate. Cool in running water to the ordinary temperature, transfer to a 1 000 ml volumetric flask using water, and dilute to the mark with water. Seal it hermetically and store.
- i) **Xylenol orange solution** Dissolve 0.1 g of xylenol orange in 100 ml of water. Store in a cool and dark place.
- j) **Buffer solution (pH5.8)** Dissolve 100 g of ammonium acetate in 700 ml of water, bring to pH5.8 by adding acetate, then dilute to 1 L with water.

5.3.3 Chemical analysis sample

In accordance with 5.2.4.

5.3.4 Sample mass

The mass of sample to be weighed out shall be 2.0 g, accurate to 1 mg.

5.3.5 Operation

The operation shall be as follows.

- a) **Preparation of sample solution** Prepare the sample solution in accordance with a) to g) of 5.2.6.1, and dissolve the precipitated salts with 5 ml of hydrochloric acid (1+1) [5.3.2 a)] and a small amount of water. Heat to boil for 1 min to 2 min, cool in running water to the ordinary temperature, transfer to a 100 ml volumetric flask using water, and dilute to the mark with water.
- b) **Masking of coexisting ions**, as follows.
 - 1) Take a 20 ml aliquot of the solution prepared according to a) in a conical beaker (300 ml), add 20 ml of disodium dihydrogen ethylenediaminetetraacetate solution (EDTA2Na solution) [5.3.2 g)], and shake well. Bring the solution to pH5 to pH6 with ammonia solution (1+2) [5.3.2 b)], and add 15 ml of the buffer solution (pH 5.8) [5.3.2 j)].

- c) **Titration**, as follows.

- ### 5.3.6 Blank test

5.3.7 Calculation

$$Al = \frac{a \times 0.002698 \times \frac{500}{50} \times \frac{100}{20}}{W} \times 100 \dots\dots\dots (2)$$

0.002 698 : conversion constant indicating the mass of aluminium corresponding to 1 ml of 0.1 mol/L zinc titrant (g/ml)

5.4.1 Summary

b) **Water volume conversion method** Hydrogen gas is introduced into an airtight container filled with water and the amount of water displaced by the gas is determined.

The reagents shall be as follows.

- a) **Hydrochloric acid**
- b) **Hydrochloric acid (1+1, 3+7)**
- c) **Aluminium powder**, having a purity of not less than 99.5 % (mass fraction) and a particle size of not more than 200 μm .
- d) **Aluminium foil**, having an Al content of 99.30 % or more specified in **JIS H 4160** or that having an equivalent quality.
- e) **Absorbing solution** Sulfuric acid (1+11)

5.4.3 Instruments and apparatus

5.4.3.1 Gas meter method

The instruments and apparatus shall be as follows. See Figure 1 for an example of the assembly of these instruments and apparatus.

- a) **Erlenmeyer flask for reaction** An Erlenmeyer flask (500 ml) made of borosilicate glass
- b) **Funnel with stopcock**
- c) **Accompanying gas absorption flask** An Erlenmeyer flask (500 ml) made of borosilicate glass
- d) **Generated gas cooling device** For example, a gas washing bottle (Walter type)
- e) **Gas meter** A wet type gas meter for experiment (5 L/h to 300 L/h, and 0.005 L in minimum reading)
- f) **Rubber tube**, suitable for connecting to the gas meter.
- g) **Rubber stopper with hole**

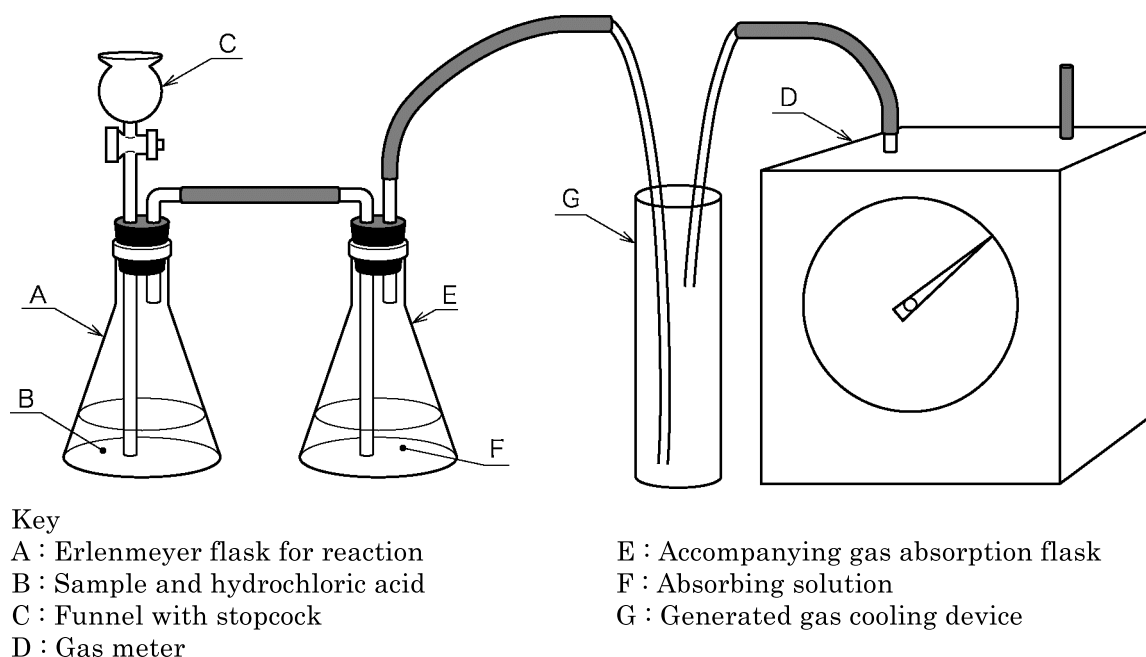
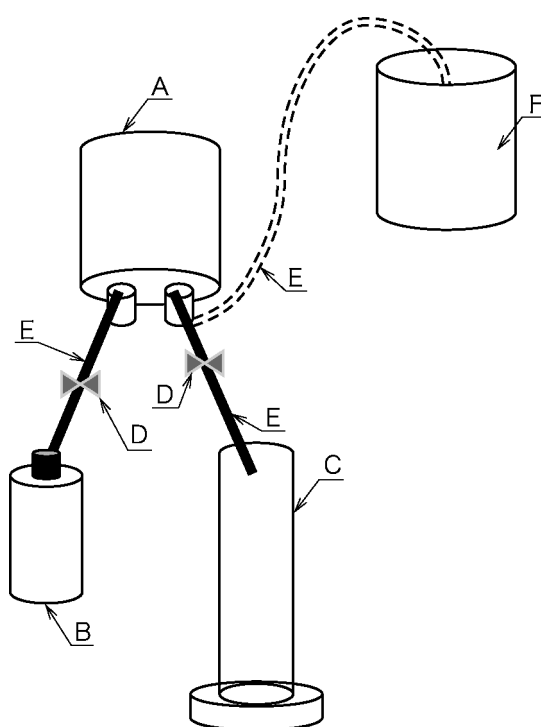


Figure 1 Example of construction of apparatus for gas meter method

5.4.3.2 Water volume conversion method

The instruments and apparatus shall be as follows. See Figure 2 for an example of the assembly of these instruments and apparatus.

- a) **Woulff bottle** (capacity 5 L)
- b) **Reaction vessel** (wide mouth)
- c) **Measuring cylinder**, with 2 000 ml capacity, used for receiving the water overflowing from the Woulff bottle.
- d) **Rubber tube**, suitable for connecting to the equipment.
- e) **Pinch valve**
- f) **Water storage tank** (for supply of water), 5 L in capacity, set up at a higher level than the Woulff bottle.
- g) **Rubber stopper with hole**



Key

A : Woulff bottle
B : Reaction vessel
C : Measuring cylinder

D : Pinch valve
E : Rubber tube
F : Water storage tank

Figure 2 Example of construction of apparatus for water volume conversion method

5.4.4 Chemical analysis sample

In accordance with 5.2.4.

5.4.5 Sample mass

The mass of sample to be weighed out in the case of gas meter method shall be 2.0 g to 5.0 g, depending on the expected metallic aluminium content of the sample (Table 1), and accurate to 1 mg, and in the case of water volume conversion method, 1.0 g and accurate to 1 mg.

5.4.6 Operation

5.4.6.1 Gas meter method

The operation shall be as follows.

- a) Weigh out a sample and transfer to an Erlenmeyer flask for reaction. Add 100 ml of hydrochloric acid (1+1) [5.4.2 b)] or hydrochloric acid (3+7) [5.4.2 b)], and quickly stopper the flask with a rubber stopper mounted with a funnel with stopcock, making sure that the stopcock is closed. About 9.5 ml of hydrochloric acid [5.4.2 a)] [approx. 36 % (mass fraction)] is required to dissolve 1 g of metallic aluminium. To prevent possible hazards, Table 1 shall be referred to for selecting the sample mass, and the concentration and volume of hydrochloric acid added.
- b) Leave to stand until bubbling of gas has ceased, and then give the vessel a light swirl to make sure there is no remaining gas.
- c) Add further 10 ml of hydrochloric acid to complete the gas generation reaction.
- d) After removing the generated gas other than hydrogen, i.e. accompanying gas, by passing through the absorbing solution [5.4.2 e)] in an accompanying gas absorption flask, cool the gas temperature by means of a generated gas cooling device until the hydrogen gas temperature stabilizes, and send the hydrogen gas to the gas meter. Throughout this procedure, the absorbing solution shall be maintained at acidity and controlled to the ordinary temperature ($20\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$), as the temperature and pH control of the absorbing solution are critical for the success of this procedure. Further, the amount of absorbing solution to be placed in the accompanying gas absorption flask shall be roughly 250 ml to 350 ml.
- e) Measure the amount of hydrogen gas with the gas meter, and at the same time, measure the temperature of hydrogen gas with the thermometer installed in the gas meter.
- f) Repeat the procedures a) to d) three times, and for each measurement, obtain the measured value to two decimal places according to Formula (8). Round the mean value of three results to one decimal place and record it as the measured value of this test.

Table 1 Sample mass and concentration of hydrochloric acid used

Expected content of metallic aluminium [% (mass fraction)]	Sample mass g	Concentration of hydrochloric acid used	Amount of hydrochloric acid used ml
5 % or over to and excl. 20 %	5.0	Hydrochloric acid (1+1)	100
20 % or over to and excl. 40 %	3.0	Hydrochloric acid (1+1)	100
40 % or over to and excl. 60 %	3.0	Hydrochloric acid (3+7)	100
60 % or over to and excl. 70 %	2.0 to 3.0	Hydrochloric acid (3+7)	100
70 % or over	2.0 to 3.0	Hydrochloric acid (3+7)	100

5.4.6.2 Water volume conversion method

The operation shall be as follows.

- Install a Woulff bottle with the rubber tube insertion inlets facing down, and connect a rubber tube each to the suction inlet for connecting the gas introduction tube and to the water discharge outlet. Set up a water storage tank at a higher level than the Woulff bottle.
- Put a sample weighed out and 50 ml of hydrochloric acid (1+1) [5.4.2 b)] in a reaction vessel. Close the mouth of the vessel with a rubber stopper, and connect to it the rubber tube extending from the suction inlet of the Woulff bottle. If the expected metallic aluminium content of the aluminium dross under test is high, hydrochloric acid (3+7) [5.4.2 b)] may be used to prevent flying hazard of rubber stopper or other associated risks.
- When the reaction with hydrochloric acid [5.4.2 a)] has started, open the pinch valve of the rubber tube connected to the suction inlet of the Woulff bottle to introduce the reaction gas into the Woulff bottle. Also open the pinch valve of the rubber tube connected to the discharge outlet of the Woulff bottle to allow the water displaced by the reaction gas to flow into the measuring cylinder.
- Continue dissolution until bubbling of gas has ceased, normally for about 5 min to 10 min at room temperature.
- After the reaction is complete, close all the pinch valves. Measure the volume of water in the measuring cylinder, and put back the water into the water storage tank.
- Record the room temperature as the temperature of the generated gas.
- Pull out the discharge-side rubber tube connecting the measuring cylinder and the Woulff bottle from the measuring cylinder and connect to the water storage tank. Open the pinch valve to allow the water in the storage tank to siphon back to the Woulff bottle, and maintain the water volume in the Woulff bottle at a constant

level. Close the pinch valve of the discharge-side rubber tube, pull out the tube from the water storage tank and connect to the measuring cylinder. Stopper the reaction bottle with a rubber stopper, and open the pinch valve of both the suction-side and discharge-side rubber tubes to allow discharge of water so that the pressure balance of the Woulff bottle and rubber tubes is brought back to the test condition. When the water discharge is complete, close the pinch valves of the rubber tubes and record the volume of water discharge received in the measuring cylinder.

- h) Repeat the procedures of a) to f) several times. Use the mean value of water volume measurements as the generated gas volume for calculation of metallic aluminium content.

5.4.7 Definition and calculation of conversion factor

5.4.7.1 Definition of conversion factor

The conversion factor F_1 is defined as the ratio between the theoretical volume and actually generated volume of hydrogen gas.

5.4.7.2 Calculation of conversion factor

The theoretical volume of hydrogen gas generated in dissolution of metallic aluminium with hydrochloric acid, which is calculated using standard conditions, cannot be directly compared with the actually measured volume which is based on other conditions than standard conditions. For the calculation of the conversion factor F_1 therefore, correction for making the temperature condition equal is necessary.

The temperature correction factor F_0 is defined by Formula (3), the temperature of the generated hydrogen gas being t (°C) :

$$F_0 = 1 + \frac{t}{273} \dots\dots\dots (3)$$

When the temperatures of hydrogen gas generated when dissolving reference material and when dissolving aluminium dross for iron and steel making are different, these temperatures shall be t_1 (°C) and t_2 (°C), respectively, and the temperature correction factors shall be F_{01} and F_{02} , respectively.

NOTE The relationship between these symbols is defined by the following formulae :

$$F_{01} = 1 + \frac{t_1}{273}$$

$$F_{02} = 1 + \frac{t_2}{273}$$

where, F_0 : temperature correction factor by temperature of generated hydrogen gas
 F_{01} : temperature correction factor by temperature of generated hydrogen gas at the time of measurement of aluminium powder [5.4.2 c)] or aluminium foil [5.4.2 d)]
 F_{02} : temperature correction factor by temperature of generated hydrogen gas

drogen gas at the time of measurement of aluminium dross for iron and steel making

t : temperature of generated hydrogen gas in gas meter at the time of measurement (°C)

t_1 : mean temperature of generated hydrogen gas at the time of dissolution of aluminium powder or aluminium foil (°C)

t_2 : mean temperature of generated hydrogen gas at the time of dissolution of aluminium dross for iron and steel making (°C)

Where the theoretical volume of hydrogen gas generated from 1 g of aluminium under standard condition is V_{01} (L), the measured volume of hydrogen gas generated in dissolution of reference materials such as aluminium powder or aluminium foil with hydrochloric acid is V_1 (L) and the temperature is t_1 , the conversion factor F_1 for these reference materials is defined by Formula (4) :

$$F_1 = \frac{V_{01}}{V_1} \dots\dots\dots (4)$$

Where the purity of reference material is P_0 % (mass fraction), the mean value of sample mass is W_1 (g) and the temperature of generated hydrogen gas is t_1 (°C), the theoretical volume of hydrogen gas V_{01} at t_1 (°C) is defined by Formula (5) which is obtained by converting the standard condition to the actually measured temperature using Formula (3) :

$$V_{01} = \frac{33.62 \times W_1}{26.98} \times \frac{P_0}{100} \times F_{01} \dots\dots\dots (5)$$

Therefore, the ratio between the theoretical volume of hydrogen gas and measured volume of generated hydrogen gas of reference material under the conditions of actual measurement, i.e. the conversion factor F_1 , is defined by Formula (6), based on Formula (4) and Formula (5) :

$$F_1 = \frac{V_{01}}{V_1} = \frac{33.62 \times W_1}{26.98 \times V_1} \times \frac{P_0}{100} \times F_{01} \dots\dots\dots (6)$$

- where,
- F_1 : conversion factor to the theoretical volume of hydrogen gas by aluminium powder or aluminium foil at 1 Pa
 - P_0 : purity of aluminium powder or aluminium foil [% (mass fraction)]
 - V_1 : measured volume of hydrogen gas generated at the time of dissolution of aluminium powder or aluminium foil (L)
 - F_{01} : temperature correction factor by temperature of hydrogen gas at the time of dissolution of aluminium powder or aluminium foil
 - W_1 : amount of aluminium powder or aluminium foil weighed out (g)
 - 26.98 : molar mass of aluminium atom (g/mol)
 - 33.62 : volume of generated hydrogen gas corresponding to 1 mol of aluminium under standard condition (0 °C and 1 atm) (L/mol)

5.4.7.3 Calculation of conversion factor F_1

Weigh two or more 1 g portion of aluminium powder [5.4.2 c)] or aluminium foil [5.4.2 d)] as the reference material to the nearest 0.1 mg, and take the mean value of the masses as W_1 (g).

Perform the measurement specified in 5.4.6 several times and use the mean value of the measured volume V_1 and temperature t_1 of generated hydrogen gas obtained in the measurements to calculate the conversion factor F_1 by Formula (6).

5.4.8 Calculation

5.4.8.1 General

Calculate the metallic aluminium content of the sample [% (mass fraction)] according to 5.4.8.2 or 5.4.8.3.

5.4.8.2 Gas meter method

The calculation used for gas meter method shall be as follows.

- a) The conversion factor F_1 [Formula (4)] is valid and equal for the measured volume of hydrogen gas in aluminium dross as well as for a reference material. Therefore, Formula (6) can be converted to Formula (7) by replacing the measured volume V_1 of generated hydrogen gas with V_2 , the mass W_1 with the sample mass W_2 , the purity [% (mass fraction)] of reference material with the metallic aluminium content P_1 [% (mass fraction)] of aluminium dross, and the temperature t_1 of generated hydrogen gas with t_2 :

$$F_1 = \frac{V_{02}}{V_2} = \frac{33.62 \times W_2}{26.98 \times V_2} \times \frac{P_1}{100} \times F_{02} \dots\dots\dots (7)$$

Transforming Formula (7) for the unknown P_1 results in Formula (8) :

$$P_1 = \frac{26.98 \times 100 \times F_1 \times V_2}{33.62 \times W_2 \times F_{02}} = 80.25 \times \left(\frac{F_1 \times V_2}{W_2 \times F_{02}} \right) \dots\dots\dots (8)$$

- where,
- P_1 : metallic aluminium content of sample [% (mass fraction)]
 - V_2 : volume of generated hydrogen gas at the time of dissolution of sample of aluminium dross for iron and steel making (L)
 - F_{02} : temperature correction factor by temperature of generated hydrogen gas at the time of measurement of sample of aluminium dross for iron and steel making according to Formula (3)
 - F_1 : conversion factor by reference material aluminium powder or aluminium foil obtained according to Formula (6)
 - W_2 : mass of sample of aluminium dross for iron and steel making (g)

Using the conversion factor F_1 obtained by Formula (6) in 5.4.7.3, calculate the metallic aluminium content P_1 of the sample by Formula (8), round the result to one decimal place, and record it as the measured value.

NOTE The gas meter method inevitably contains a certain amount of error due to being an indirect method; the following method can be used to verify the reliability of the measured value of metallic aluminium content P_1 of

the sample obtained by Formula (8).

- a) Analyze the same sample by the reference method of **5.2** or **5.3** at the same time as the analysis by the gas meter method for the purpose of preventing oxidation, and compare the two results to verify the reliability of the measured value by the gas method.
- b) If the relative error between the two results is larger than 2 %, examine if each of the processes specified in **5.4.6.1** is faithfully carried out, and review the sample pretreatment method, especially the number of pulverizations immediately before testing and the aperture size of the sieve used.
- c) Increasing the number of pulverizations and decreasing the sieve aperture size will reduce the error relative to the reference measured value by **5.2** or **5.3** to less than 1.0 %.

5.4.8.3 Water volume conversion method

The calculation used for the water volume conversion method shall be as follows.

- a) Calculate the metallic aluminium content of the sample [% (mass fraction)] according to Formula (8) using the water volume value obtained by subtracting the water volume recorded according to **5.4.6.2 g)** from the discharged water volume by hydrogen gas generation measured according to **5.4.6.2 e)** as the measured volume of generated hydrogen gas, and using the room temperature as the temperature t_1 and t_2 of generated hydrogen gas since these temperature cannot be measured.
- b) Since the water conversion method has more error factors than the gas meter method, the metallic aluminium content P_1 of the sample obtained by **a)** needs to be corrected using the measured value of the gas meter method (**5.4.8.2**) according to the following method.
 - 1) Perform measurement by the gas meter method and that by the water volume conversion method on the same sample.
 - 2) Plot the measured values of the gas meter method on the vertical axis and the measured values of the water volume conversion method on the horizontal axis to create a regression line. Use this regression line to convert the value by the water volume conversion method to the value by the gas meter method, and round the result to one decimal place.

5.5 Measurement using reaction solution temperature

5.5.1 Summary

The sample is reacted with hydrochloric acid, and the heat generated in the reaction is used to heat the reaction solution. The difference between the reaction solution temperature before and after the reaction is used to calculate the metallic aluminium content.

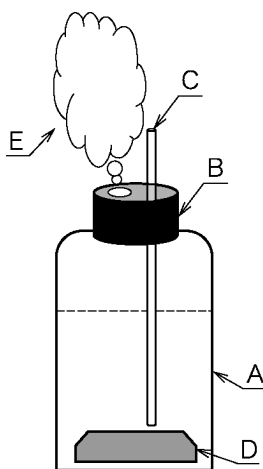
5.5.2 Reaction solution

Use hydrochloric acid (1+2) previously brought to $30\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ by hot water bath or water cooling using a container capable of minimizing volatilization of hydrochloric acid.

5.5.3 Instruments

The instruments shall be as follows. See Figure 3 for an example of structure of the apparatus.

- a) **Thermometers**, specified in **JIS Z 8710**, and as follows.
 - 1) A platinum or nickel resistance thermometer with the measuring range of $0\text{ }^{\circ}\text{C}$ to $100\text{ }^{\circ}\text{C}$ and the minimum reading of $0.1\text{ }^{\circ}\text{C}$, accurate to $\pm 0.05\text{ }^{\circ}\text{C}$ of the measured value, and having been calibrated.
 - 2) A mercury-in-glass thermometer of precision type.
- b) **Dewar bottle**, large enough to contain the reaction solution without overflowing during the reaction.
- c) **Hot water bath**, capable of controlling the water temperature close to $30\text{ }^{\circ}\text{C}$ with the accuracy of $\pm 1\text{ }^{\circ}\text{C}$.
- d) **Rubber stopper with hole**



Key

A : Dewar bottle

B : Rubber stopper with hole

C : Thermometer

D : Dross for reaction

E : Generated gas

Figure 3 Example of apparatus structure for measurement using reaction solution temperature

5.5.4 Chemical analysis sample

In accordance with 5.2.4.

5.5.5 Sample mass