

JIS

JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS G 1317 : 1998

Method for chemical analysis of ferromolybdenum

ICS 77.100

Descriptors : ferromolybdenum, ferro-alloys, molybdenum-containing alloys,
chemical analysis and testing, testing, determination of content,
molybdenum, carbon, silicon, phosphorus, sulphur, copper, aluminium

Reference number : JIS G 1317 : 1998 (E)

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS G 1317: 1982** is replaced with **JIS G 1317: 1998**.

In this revision, **ISO 4173** has been employed in Annex 2 for conforming to International Standards, and determinations of each component are specified in Annexes 1 to 14.

Date of Establishment: 1953-03-28

Date of Revision: 1998-02-20

Date of Public Notice in Official Gazette: 1998-02-20

Investigated by: Japanese Industrial Standards Committee
Divisional Council on Iron and Steel

JIS G 1317:1998, First English edition published in 1999-04

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

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Printed in Japan

Method for chemical analysis of ferromolybdenum

Introduction In the application of standard, normative references to the standard shall be simultaneously referred to. Since ferromolybdenum has many chances to be used as the supplementary raw material in iron and steel making process, JIS G 1317, which specifies the determination of molybdenum as main element and other 6 elements containing carbon which give rise to discussion in the iron and steel production, was established in 1953. Annex 2 is the Japanese Industrial Standard prepared based on ISO 4173 *Ferromolybdenum—Determination of molybdenum content—Gravimetric method*, published in 1980 as the first edition, without modifying technical contents.

The “Informative reference” underlined with dots in Annex 2 shows the matters not given in the original International Standard.

1 Scope This Standard specifies the methods for determination of molybdenum, carbon, silicon, phosphorus, sulfur, copper and aluminum in ferromolybdenum.

2 Normative references The following standards contain provisions which, through reference in this standard, constitute provisions of this Standard. The most recent editions of the standards indicated below shall be applied.

JIS G 1301 *General rules for chemical analysis of ferroalloys*

JIS K 0116 *General rules for atomic emission spectrometry*

JIS K 0121 *General rules for atomic absorption spectrochemical analysis*

JIS K 8001 *General rule for test methods of reagents*

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

JIS Z 2616 *General rules for determination of sulfur in metallic materials*

JIS Z 8402 *General rules for permissible tolerance of chemical analyses and physical tests*

3 General matters General matters common to the methods for chemical analysis shall be in accordance with JIS G 1301, JIS K 0116 and JIS K 0121. However, these shall not be applied to Annex 2.

4 Classification of the method for determination The methods for determination of each component in ferromolybdenum shall be according to Table 1.

Table 1 The methods for determination of each component in ferromolybdenum

Component	Determination method	Range of percentage of components % (m/m)	Annex (normative) No.
Molybdenum	Iron separation zinc amalgam reduction potassium permanganate titration method	50 or over up to and incl. 80	1
	8-quinolinol molybdenum gravimetric method (ISO 4173)	50 or over up to and incl. 80	2
Carbon	Combustion-gas volumetric method	0.05 or over up to and incl. 6.0	3
	Combustion-infrared absorption method	0.001 or over up to and incl. 6.0	4
Silicon	Silicon dioxide gravimetric method	0.05 or over up to and incl. 3.0	5
	ICP emission spectrochemical method	0.02 or over up to and incl. 1.0	6
Phosphorous	Beryllium hydroxide coprecipitation separation phosphomolybdenum blue absorptiometric method	0.002 or over up to and incl. 0.10	7
	Beryllium hydroxide coprecipitation separation ICP emission spectrochemical method	0.002 or over up to and incl. 0.10	8
Sulfur	Combustion-potassium iodate titration method	0.005 or over up to and incl. 0.20	9
	Combustion-infrared absorption method	0.001 or over up to and incl. 0.20	10
Copper	Atomic absorption method	0.01 or over up to and incl. 0.50	11
	ICP emission spectrochemical method	0.01 or over up to and incl. 0.50	12
Aluminium	Atomic absorption method	0.005 or over up to and incl. 0.20	13
	ICP emission spectrochemical method	0.005 or over up to and incl. 0.20	14

Annex 1 (normative)**Determination of molybdenum — Iron separation zinc amalgam reduction
potassium permanganate titration method**

1 Summary A sample is decomposed with sulfuric acid and nitric acid, concentrated by heating. Remove nitric acid by generation of white fumes of sulfuric acid, and separate iron, etc. as hydroxide with sodium hydroxide. Concentration of acid is adjusted with sulfuric acid, add zinc amalgam and mix by shaking. After reducing molybdenum, it is titrated with potassium permanganate standard solution.

2 Reagents The reagents shall be as follows :

- a) **Nitric acid (1+1)**
- b) **Sulfuric acid (1+1)**
- c) **Carbon dioxide**
- d) **Fusing mixture [sodium carbonate 1 part and potassium carbonate 1 part]**
- e) **Zinc amalgam** 450 g of mercury is transferred into an Erlenmeyer flask (300 ml), added with 10 g of granular zinc, from which oxide film on the surface is removed with sulfuric acid (1+10), and its surface is covered with small amount of sulfuric acid (1+10). The granular zinc is dissolved by heating on water bath with occasional shaking, cooled to room temperature and the surface of amalgam is covered with small amount of sulfuric acid (1+10) to be stored.

WARNING FOR SAFETY As zinc amalgam is heavily poisonous, this reagent and a solution with this reagent added shall be handled with care.

- f) **Sodium hydroxide solution (200 g/l)**
- g) **Ammonium iron (III) sulfate solution** Dissolve 100 g of ammonium iron (III) sulfate 12 hydrate in water, add little by little 50 ml of sulfuric acid (1+1) and 400 ml of phosphoric acid under mixing it by shaking, and dilute to 1 000 ml with water.
- h) **0.02 mol/l potassium permanganate standard solution** The method for preparation and standardization shall be in accordance with 4.5 (7) of JIS K 8001.

3 Apparatus The reduction apparatus shown in Attached Fig. 1 of Annex 1 shall, as a rule, be used. The part below the lower cock shall be filled with water which is boiled to remove air and cooled.

4 Test portion Weigh out 0.50 g to the nearest 0.1 mg.

5 Operation

5.1 Decomposition of sample The decomposition of a sample shall be carried out according to the following procedures.

- a) Weigh out a sample and transfer it to a beaker (300 ml) and cover it with a watch glass.
- b) Add 20 ml of sulfuric acid (1+1) and 5 ml of nitric acid (1+1), and decompose it by

heating.

- c) After allowing it to cool, wash the bottom side of the watch glass, remove it and heat the beaker until white fumes of sulfuric acid start to be generated.
- d) Expel nitric acid thoroughly by further heating for (15 to 20) minutes under generating white fumes of sulfuric acid.
- e) After allowing it to cool, add about 100 ml of warm water and dissolve soluble salts by heating ⁽¹⁾.
- f) Filter it through a filter paper (Class 5B), wash it with warm water, receive the filtrate and the washings with a beaker (300 ml) and reserve it as a main solution.

Transfer the residue and filter paper into a nickel crucible (No.30), incinerate the filter paper at 600 °C max. Then add fusing mixture [2 d)] about 10 times amount of the residue and dissolve by heating. After allowing it to cool, dissolve the melt in water and compound it into main solution.

Note ⁽¹⁾ In case of absence of insoluble residue, the following operation f) shall not be carried out.

5.2 Separation of iron, etc. Separation of iron, etc. shall be carried out according to the following procedure.

- a) Add little by little sodium hydroxide solution to the solution obtained in 5.1 e) or f) with stirring, neutralize it until the precipitate of iron (III) hydroxide is slightly produced and further add 20 ml of it in excess.
- b) After boiling it for (3 to 5) minutes by gentle heating, cool it to ordinary temperature, transfer it into a volumetric flask of 250 ml by adding water and dilute it with water up to the marked line.
- c) Filter the solution into a dry beaker (300 ml) through a dry filter paper (Class 5B).
- d) Discard the initial filtrate, take separately 50 ml from the subsequent filtrate and transfer it into a beaker (100 ml).

5.3 Reduction of molybdenum Reduction of molybdenum shall be carried out according to the following procedure.

- a) Add 6 ml of sulfuric acid (1+1) to the solution which is separately taken in 5.2 d) and stir it.
- b) Transfer the solution into reduction apparatus of 3 with about 200 g of zinc amalgam [2 e)] in it by washing with a small amount of water ⁽²⁾.
- c) After substituting air by passing carbon dioxide for about 3 minutes, mix them by violently shaking for about 5 minutes and reduce molybdenum thoroughly.

Note ⁽²⁾ As the concentration of sulfuric acid at reduction operation, approximately 0.75 mol/l should be suitable, so the amount of water for transferring shall be smaller than 15 ml.

5.4 Titration Carefully and thoroughly transfer the zinc amalgam in a reduction apparatus to the receiver, titrate it immediately with 0.02 mol/l potassium permanganate standard

solution [2 h)], taking care that the air does not enter the reduction apparatus ⁽³⁾ ⁽⁴⁾, and take the point at which color of solution turns to faint red as the end point.

Notes⁽³⁾ When the reduction apparatus shown in Attached Fig. 1 (b) in Annex 1 is used, it shall be titrated with passing carbon dioxide.

⁽⁴⁾ As the reduced molybdenum is relatively easy to be oxidized, it shall be carefully operated so that the solution does not touch the air after reduction of molybdenum.

Further, in order to prevent oxidation of molybdenum, the following may be carried out : reduce molybdenum and remove zinc amalgam from the solution, add ammonium iron (III) sulfate solution [2 g] and oxidize molybdenum, and titrate the reduced iron (II).

6 Blank Test Blank test shall not be carried out.

7 Calculation The content of molybdenum in sample shall be calculated from the following formula.

$$Mo = \frac{V \times F \times 0.003\,198}{m \times \frac{1}{5}} \times 100$$

where *Mo* : content of molybdenum in the sample [% (m/m)]

V : titration volume of 0.02 mol/l potassium permanganate standard solution obtained in 5.4 (ml)

F : factor of 0.02 mol/l potassium permanganate standard solution

m : test portion (g)

8 Tolerance Tolerance ⁽⁵⁾ shall be in accordance with Table 1 of Annex 1.

Annex 1 Table 1 Tolerance

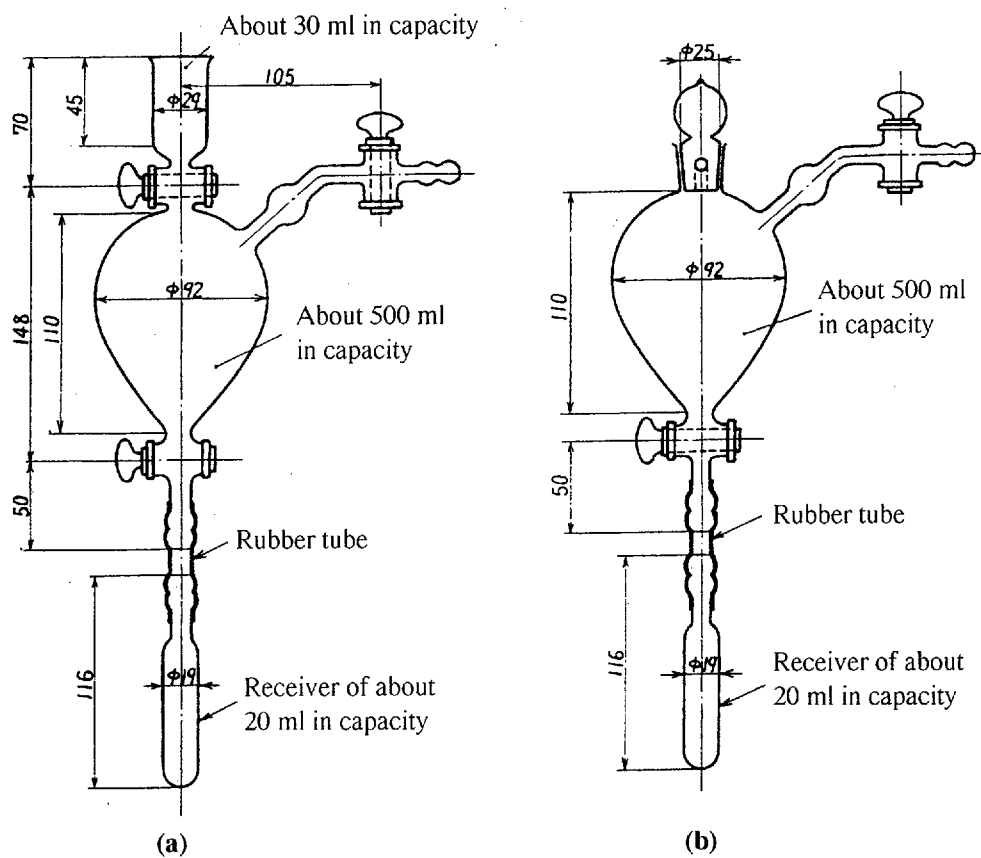
Unit : % (m/m)

Alloy type	Tolerance of reproducibility within laboratory	Tolerance of reproducibility between laboratories
Low carbon ferromolybdenum	<i>D</i> (<i>n</i>) [0.14]	<i>D</i> (<i>n</i>) [0.18]
High carbon ferromolybdenum	<i>D</i> (<i>n</i>) [0.15]	<i>D</i> (<i>n</i>) [0.32]

Note ⁽⁵⁾ *D* (*n*) in calculation formula of tolerance means *D* (*n*, 0.95) and this value is according to Table 4 of JIS Z 8402. The *n*-value in tolerance of reproducibility within laboratory means the number of analyses in the same test laboratory and that in tolerance of reproducibility between laboratories means the number of test laboratories which carried out the analysis.

Informative reference : These tolerances are determined by the results of cooperation test using low carbon ferromolybdenum of 60.8 % (m/m) molybdenum and high carbon ferromolybdenum of 60.4 % (m/m) molybdenum.

Unit : mm



Annex 1 Attached Fig. 1 Reduction apparatus

Annex 2 (normative)
Determination of molybdenum — 8-quinolinol
molybdate gravimetric method (ISO 4173)

1 Scope This Annex 2 specifies the method for determination of molybdenum in ferromolybdenum by 8-quinolinol molybdate gravimetric method.

This method shall be applied to ferromolybdenum in which molybdenum content is 50 % (m/m) or over up to and including 80 % (m/m).

Informative reference 1 In the original Standard, the name of this method is described as only “gravimetric method”. However, in this Standard, it is stated as “8-quinolinol molybdate gravimetric method” in accordance with the name of determination method in JIS.

The name of reagent in the original ISO Standard is described as “8-hydroxyquinoline”, but in this Standard “8-quinolinol” is used in accordance with nomenclature of IUPAC.

2 In the original ISO Standard, it is said as “molybdenum contents in the range normally found in ferromolybdenum”, but definite figures are given in this Standard in accordance with ranges of concentration of molybdenum specified in ferromolybdenum specification.

2 Normative references

ISO 3713 *Ferroalloys—Sampling and preparation of samples—General rules* [foot-note¹⁾]

Informative reference : In the original ISO Standard, the footnote says “At present at the stage of draft”, but this Standard was published in 1987.

3 Principle Dissolve the test portion, and separate iron with sodium hydroxide. Precipitate the molybdenum as the 8-quinolinol salt in the presence of ethylene diamine tetraacetate dihydrogen disodium dihydrate (hereafter referred to as EDTA2Na) and ammonium oxalate. Filtrate, dry and weigh anhydrous 8-quinolinol molybdate [$\text{MoO}_2(\text{C}_8\text{H}_7\text{NO})_2$].

4 Reagents During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Sulfuric acid 50 % (V/V) solution Add little by little 500 ml of sulfuric acid (ρ 1.84 g/ml) into 400 ml of water with cooling. Cool it with stirring and dilute the volume to 1 000 ml with water.

4.2 Nitric acid 25 % (V/V) solution Dilute 250 ml of nitric acid (ρ 1.42 g/ml) to 1 000 ml with water.

4.3 Hydrofluoric acid (ρ 1.14 g/ml)

4.4 Hydrochloric acid 50 % (V/V) solution Add water into 500 ml of hydrochloric acid (ρ 1.16 g/ml to 1.18 g/ml) and dilute the volume to 1 000 ml with water.

4.5 Sodium hydroxide solution (460 g/l to 480 g/l) Weighing (460 to 480) g of sodium hydroxide, put it into a polyethylene beaker. Dissolve sodium hydroxide in water by cooling the outside wall of the beaker with flowing water and dilute to 1 000 ml and mix. This solution shall be stored in a polyethylene bottle.

4.6 EDTA2Na solution (10 g/l) Dissolve 10 g of EDTA2Na into water and dilute to 1 000 ml.

4.7 Ammonia solution 50 % (V/V) Add water into 500 ml of ammonia solution (ρ 0.91 g/ml) and dilute the volume to 1 000 ml.

4.8 8-quinolinol solution (30 g/l) neutral solution Dissolve 3 g of 8-quinolinol in 12 ml of acetic acid, add 60 ml of water and warm to about 40 °C. Add the ammonia solution (4.7) dropwise until a slight permanent precipitate is formed. Dissolve again the precipitate by adding dropwise acetic acid with care, cool and dilute to 100 ml with water.

4.9 Nitric acid ρ 1.42 g/ml

4.10 Hydrochloric acid ρ 1.16 g/ml to 1.18 g/ml

4.11 Ammonium oxalate

5 Apparatus Use the usual apparatus and equipment in an analysis laboratory.

6 Sample Use powder sample which will pass through a sieve with a mesh size of 160 μm , prepared in accordance with ISO 3713.

7 Operation

7.1 Test Portion Owing to the inhomogeneity of ferromolybdenum, the sample which has been passed through a screen of mesh opening 160 μm should be sieved again with a screen of mesh opening 75 μm to 80 μm and proportionate parts of over- and under-size used for analysis.

Weigh out 1 g for 75 % (m/m) ferromolybdenum or 1.25 g for 50 % (m/m) ferromolybdenum.

7.2 Determination Take the test portion (7.1) in a polytetrafluoroethylene (PTFE) beaker (150 ml), add 10 ml of sulfuric acid solution (4.1) and warm without boiling. Add, dropwise, the nitric acid (4.9) until the test portion is dissolved and then add 0.5 ml in excess. Add 5 ml of the hydrofluoric acid (4.3) and evaporate until fumes of sulfur trioxide are evolved. Continue fuming for 15 minutes, cool, add 5 ml each of hydrochloric acid (4.10) and nitric acid (4.9), warm for 10 minutes and dilute to about 50 ml.

Into a tall beaker (capacity 650 ml to 800 ml), take 50 ml of the sodium hydroxide solution (4.5) and 50 ml of water. Heat to boil and place a funnel in a mouth of the beaker.

Note : It is desirable to use the funnel which has been made by drawing out the stem of a 75 ml funnel to produce a jet size of diameter approximately 1 mm.

Transfer the test solution slowly, through the funnel, into the beaker containing boiling sodium hydroxide solution. Rinse the PTFE beaker with hot water (85 °C to 90 °C) and transfer the washings to the funnel. Rinse the funnel and upper surface of the tall beaker, boil the solution for 2 to 3 minutes, dilute to about 450 ml and cool.

Transfer to 500 ml of one mark volumetric flask, and dilute to the mark with water. Dry the original tall beaker, transfer the solution of the volumetric flask into it and allow to stand for 15 to 20 minutes.

Filter a portion of the solution through a dry, rapid-hardened filter paper and transfer a 100 ml aliquot to a beaker (400 ml). Dilute it to about 200 ml with water and add 10 ml of the EDTA2Na solution (4.6) and 3 g of the ammonium oxalate (4.11). Warm gently to dissolve the ammonium oxalate, cool to room temperature and adjust the solution to pH 4.0 using a pH meter by addition of the hydrochloric acid (4.4) and the ammonia solution (4.7).

Heat the solution to boiling and add 20 ml of the 8-quinolinol solution (4.8), ensuring that the solution continues to boil throughout the addition.

Note : It is essential that the precipitation be carried out in a boiling solution. If precipitation is carried out in a solution below boiling point, the precipitate formed is more difficult to wash and initially gives high recoveries ; if dried until constant mass (perhaps 2 to 3 days) is reached, low recoveries may sometimes be obtained.

Allow to stand for 5 to 10 minutes at 80 °C to 90 °C with occasional stirring and filter through a sintered glass filter (porous 5 µm to 10 µm) with a known mass (this mass shall be m_2), by using gentle suction. Remove the precipitate from the walls of beaker with using a glass rod with rubber, transfer it to the glass filter and wash the precipitate in the filter with about 100 ml of hot water (80 °C to 90 °C).

Informative reference : As it is difficult to understand the description in the original text, this writing was added.

Note : During filtration, it is needed that not more than half of the filter should be filled with the solution at any time. This procedure facilitates the efficient washing of the filter and provides a precipitate free from soluble salts.

Dry overnight at 125 °C, cool and weigh. Re-dry to constant mass and weigh as anhydrous 8-quinolinol molybdate (this mass shall be m_1).

Informative reference : As it is difficult to understand the description in the original text, this writing was added.

Note : The following procedures are recommended for handling and cooling a sintered glass filter.

- a) After washing with suction, the outside wall of the filter should be washed with hot water, dried on a filter paper and, from that stage, should not be touched with bare hands. The filters (or crucibles) should preferably be handled with tweezers or for a minimum period in a gloved hand.
- b) After drying in an oven, both the empty filters and those containing the 8-quinolinolate precipitates should be placed in an efficient desiccator and allowed to stand at room

temperature for 1 hour before weighing.

- c) Before use, the filters should be washed several times with hot water, nitric acid solution (4.2) and finally with water and dried at 125 °C. Cool in an efficient desiccator to room temperature and use this filter as a filter of which the mass is known after confirming the constant mass.

Informative reference : In order to make it easy to understand the meaning of the filter of which the mass is known, this writing was added.

Transfer the used filter in a small beaker containing 5 ml to 10 ml of sulfuric acid solution (4.1) after removing the bulk of the precipitate with a jet of water and fill it with nitric acid (4.9). Heat the beaker until fumes of sulfuric acid are generated, and cool it. Remove the filter from the beaker and pass about 100 ml of hot water through the filter under gentle suction. Carry out the washed filter as in a) and b) above.

8 Expression of results The molybdenum content [% (m/m)] is calculated by the following formula.

$$\frac{(m_1 - m_2) \times 23.05}{m_0}$$

where m_1 : the mass of the glass filter containing the anhydrous 8-quinolinol molybdate (g)

m_2 : the mass of the used glass filter (g) [Note : Refer to c)]

m_0 : the mass of the test portion in the final aliquot solution (g)

Remarks : As it is difficult to understand the description in the original text, this writing was added.

9 Reproducibility A cooperated test of this method was carried out by different seven laboratories. The tests of five times for each of two samples were carried out by each analyst. From the results obtained, the 95 % confidence limits (2s) given in the table have been calculated.

Table Reproducibility according to alloy types

Alloy type	Molybdenum content % (m/m)	Standard deviation		Tolerance of reproducibility between laboratories index 2s
		S_b	S_w	
50 % (m/m) Ferromolybdenum	50.33	0.081	0.090	0.24
75 % (m/m) Ferromolybdenum	75.34	0.085	0.100	0.26

10 Test report The following particulars shall be included in a test report.

- The items in accordance with this Annex
- The items on identification of sample

- c) The results of analysis
- d) Any unusual features noted during the determination
- e) The detailed items of any operation unspecified in this Annex or optional operation

Annex 3 (normative)**Determination of carbon — Combustion-gas volumetric method**

1 Summary Heat a sample in an oxygen gas flow at a high temperature, oxidize carbon to carbon dioxide. Collect it together with oxygen into a burette, measure the volume of gas. Then let an alkali solution absorb carbon dioxide to be removed, and measure the volume of remaining gas.

2 Apparatus The apparatus shall be in accordance with 6.3.2 of 6.3 in JIS Z 2615.

3 Equipment and materials The equipment and materials shall be in accordance with 5 in JIS Z 2615.

4 Test portion and additional weight of accelerator Weigh out the sample, to the nearest 0.1 mg, according to Annex 3 Table 1:

Annex 3 Table 1 Test portion

Carbon contents % (m/m)	Test portion g
0.01 or over to and excl. 0.1	2.0
0.1 or over to and excl. 1.0	1.0
1.0 or over to and excl. 3.0	0.50
3.0 or over up to and incl. 6.0	0.25

5 Operation

WARNING FOR SAFETY In the combustion process, it shall be taken care that a pair of tongs for a crucible shall always be used in handling of a porcelain combustion boat heated at a high temperature or a combustion crucible for fear that the skin or fingers of an operator may be burnt.

And efforts shall be made to prevent occurrence of a fire by paying attention to the treatment of excessive exhaust oxygen gas.

5.1 Preparation for operation The preparation for operation shall be in accordance with 6.3.3 of 6.3 in JIS Z 2615.

When a tubular electric resistance heating furnace is used, a temperature in the combustion tube shall be kept at 1 300 °C. to 1 400 °C ⁽¹⁾.

When a high frequency induction heating furnace is used, the conditions for high frequency induction heating shall be established ⁽²⁾.

Notes⁽¹⁾ Correct the temperature paying regard to the differences between the temperature of the inside combustion tube and that of thermometer indication.

- (²) For example, the parameter, such as anodic current of high frequency oscillator and grid current which are depending on the specification of the applied equipment.

5.2 Determination The determination shall be carried out in accordance with either one of the following procedures (³). Attached Fig. 2 (No.1) in **JIS Z 2615** shall be referred to in each case.

Note (³) In the daily works, the apparatus and equipment shall be tested whether they are controlled to satisfy the performance criteria or not, at the beginning, the middle, and the end of a working time, in accordance with the same procedures as the determination of the analytical sample, by using the samples with known carbon contents.

a) When a tubular electric resistance heating furnace is used

- 1) Weigh out the sample and transfer it into the porcelain combustion boat (⁴).
- 2) Open the inlet of the combustion tube of the tubular electric resistance heating furnace and insert the porcelain combustion boat containing the sample into the combustion tube and place it in the center of the tube. Immediately, stopper it airtightly.
- 3) Turn a three way stopcock f) to the furnace side and allow to stand for 2 min, while adjusting the flow of oxygen.
- 4) Hereafter, carry out the procedures in accordance with 6.3.4 2) to 6) of 6.3 in **JIS Z 2615**.

Note (⁴) Select the most adequate accelerator (for example iron, tin, tungsten, etc.) which is listed in the 5 (13) in **JIS Z 2615**, and add the optimum amount of accelerator for the applied apparatus or equipment. Mix the accelerator added thoroughly with the sample.

b) When a high frequency induction heating furnace is used

- 1) Weigh out the sample and transfer into the porcelain combustion crucible (⁴).
- 2) After connecting the apparatus and confirming the airtightness, close the three way stopcock (m), and open the three way stopcock (l) to the open air side.
- 3) Place the porcelain combustion crucible containing the sample obtained in 1) operation on a bearer of high frequency induction heating furnace, insert the crucible to the center of the heating coil with operating the handle and close the combustion tube.
- 4) Flow oxygen into the combustion tube and replace the air with oxygen.
- 5) Close the three way stopcock (l) and start to work the high frequency induction heating furnace.
- 6) Open the three way stopcock (m) to the furnace side after passing (10 to 20) s. Then open the three way stopcock (l) slightly to the furnace side and introduce the combustion gas into the burette (n) through the desulfurization tube (j) and the cooling tube (k).
- 7) After almost finishing the combustion of the sample which takes about 1 min, collect the mixed gas rapidly close to the bottom graduation of the burette (n).
- 8) Close the three way stopcocks (l) and (m). Allow to stand still for about 1 min after