

JIS

JAPANESE INDUSTRIAL STANDARD

Methods for Chemical Analysis of Chromium Metal

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In the event of any doubt arising,
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Methods for Chemical Analysis of
Chromium Metal

G 1323-1989

1. Scope

This Japanese Industrial Standard specifies the methods for determination of chromium, carbon, phosphorus, sulfur, iron and aluminium in chromium metal.

Remark: The units and numerical values given in { } in this Standard are based on the traditional units and are appended for informative reference.

2. General Matters

General matters common to the methods for chemical analysis shall be in accordance with JIS G 1301.

3. Method for Determination of Chromium

3.1 Method for Determination The method for determination shall not be in accordance with chemical analysis and after determination of impurities, the residue, be taken.

3.2 Calculation The chromium content in the sample shall be calculated to two places of decimals according to the following formula, and the rest shall be discarded.

$$\text{Chromium wt\%} = 100 - (C + Si + P + S + Fe + Al)$$

where, C: carbon content (wt %)

Si: silicon content (wt %)

P: phosphorus content (wt %)

S: sulfur content (wt %)

Fe: iron content (wt %)

Al: aluminium content (wt %)

Applicable Standards:

JIS G 1301-General Rules for Chemical Analysis of Ferroalloys

JIS Z 2615-General Rules for Determination of Carbon in Metallic Materials

JIS Z 2616-General Rules for Determination of Sulfur in Metallic Materials

4. Method for Determination of Carbon

4.1 Division of Methods for Determination The method for determination of carbon shall be in accordance with one of the following:

- (1) Combustion - Neutralization Titrimetric Method This method applies to the sample of 0.05 wt % or over up to and excluding 0.1 wt % in carbon content.
- (2) Combustion - Conductometric Method This method applies to the sample of 0.001 wt % or over in carbon content.
- (3) Combustion - Coulometric Method This method applies to the sample of 0.001 wt % or over in carbon content.
- (4) Combustion - Infrared Absorptiometric Method This method applies to the sample of 0.001 wt % or over in carbon content.

4.2 Combustion - Neutralization Titrimetric Method

4.2.1 Summary Heat the sample in an oxygen gas flow, oxidize the carbon sufficiently to make carbon dioxide, after collecting this together with oxygen into a burette, introduce into a certain amount of sodium hydroxide standard solution to absorb the carbon dioxide, and thereafter titrate the excess sodium hydroxide by sulfuric acid standard solution.

4.2.2 Reagents Reagents shall be in accordance with 6.4.2 of JIS Z 2615.

4.2.3 Devices and Materials Devices and materials shall be in accordance with 5. of JIS Z 2615.

4.2.4 Apparatus The apparatus shall be in accordance with 6.4.3 of JIS Z 2615.

4.2.5 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver Allow the sample to be 1.0 g. Select the ignition improver most suitable ⁽¹⁾ from those indicated in 5. (13) of JIS Z 2615, add the amount most suitable for weighing-out amount of sample and combustion apparatus to mix thoroughly or cover on the sample.

Note ⁽¹⁾ High purity iron, tin, tungsten, etc.

4.2.6 Operation

4.2.6.1 Preparatory Operation The preparatory operation shall be in accordance with 6.4.4 of JIS Z 2615.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1400 to 1450°C ⁽²⁾.

Further, in the case of using a high frequency induction heating furnace, set the conditions ⁽³⁾ relating to the high frequency induction heating furnace.

Notes ⁽²⁾ Correct by taking precautions to the difference between the temperature indicating of pyrometer and the combustion tube inside temperature.

- (3) Those mean the conditions to be determined depending upon the specifications of apparatus used such as anode current, grid current, etc. of high frequency oscillator.

4.2.6.2 Determination Operation The determination operation shall be carried out according to either one of the following:

- (1) In the Case of Using Tubular Electric Resistance Heating Furnace The operation shall be in accordance with 6.4.5 of JIS Z 2615.
- (2) In the Case of Using High Frequency Induction Heating Furnace The operation shall be in accordance with Remark 2. of 6.4 of JIS Z 2615.

4.2.7 Blank Test The blank test shall be carried out according to either one of the following:

- (1) In the Case of Using Tubular Electric Resistance Heating Furnace The blank test shall be in accordance with 6.4.6 of JIS Z 2615.
- (2) In the Case of Using High Frequency Induction Heating Furnace By using a crucible put-in with the ignition improver of the same amount as the amount of added sample without putting-in the sample, carry out the operation of 4.2.6.2 (2).

Further, in the case of using an ignition improver not to generate induction current by high frequency current, add 0.5 to 1.0 g of iron or the like of known carbon content and as lower as possible in carbon content to carry out, and correct the carbon content included in the added iron or the like to take it as the blank test value.

4.2.8 Calculation The calculation shall be in accordance with 6.4.7 of JIS Z 2615.

4.3 Combustion - Conductometric Method

4.3.1 Summary Heat the sample in an oxygen gas flow to oxidize sufficiently the carbon to carbon dioxide, allow to absorb into a certain amount of alkali solution, and measure the change of conductivity of alkali solution before and after absorption.

4.3.2 Reagents Reagents shall be in accordance with 6.6.2 of JIS Z 2615.

4.3.3 Devices and Materials Devices and materials shall be in accordance with 5. of JIS Z 2615.

4.3.4 Apparatus The apparatus shall be in accordance with 6.6.3 of JIS Z 2615 (4).

Note (4) Instead of tubular electric resistance heating furnace, a high frequency induction heating furnace may be used. In the case of using a high frequency induction heating furnace, for example the boat and boat cover are read as crucible and cover, instead of them.

4.3.5 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver Make the weighing amount of sample the most suitable amount (usually 0.5 to 1.0 g) to the using apparatus. Select the ignition improver most suitable ⁽¹⁾ from those indicated in 5. (13) of JIS Z 2615, add the most suitable amount to the weighing-out amount of sample and combustion apparatus and mix thoroughly or cover on the sample.

4.3.6 Operation

4.3.6.1 Preparatory Operation The preparatory operation shall be in accordance with 6.6.4 of JIS Z 2615.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1400 to 1450°C ⁽²⁾.

Further, in the case of using a high frequency induction heating furnace, set the conditions ⁽³⁾ relating to the high frequency induction heating furnace.

4.3.6.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The operation shall be in accordance with 6.6.5 of JIS Z 2615.

(2) In the Case of Using a High Frequency Induction Heating Furnace

- (a) Place a crucible containing the sample and an ignition improver on a receiving stand and close the combustion tube. After substituting the air in the tube by feeding-in oxygen at the designated flow rate, actuate the high frequency induction heating furnace.
- (b) When the indicating value of recorder or indicator increases gradually and a definite value is indicated, read out the indicated value and cut the switch of high frequency to take out the crucible.

4.3.7 Blank Test The blank test shall be carried out according to either one of the following:

(1) In the Case of Using Tubular Electric Resistance Heating Furnace The blank test shall be in accordance with 6.6.6 of JIS Z 2615.

(2) In the Case of Using High Frequency Induction Heating Furnace By using a crucible put-in with only ignition improver of the same amount as the amount added to the sample without putting-in the sample, carry out the operation of 4.3.6.2 (2).

Further, in the case of using such ignition improver that induction electric current is not generated by high frequency current, add 0.5 to 1.0 g of iron or the like of known carbon content and as lower as possible in carbon content to carry out, and correct the carbon amount contained in the added iron or the like to take it as the blank test value.

4.3.8 Calculation The calculation shall be in accordance with 6.6.7 of JIS Z 2615.

4.4 Combustion - Coulometric Method

4.4.1 Summary Heat the sample in oxygen gas flow to oxidize sufficiently the carbon to make carbon dioxide, allow to absorb in weak alkaline barium salt solution made at a definite pH, and measure the quantity of electricity required for returning the pH reduced by absorption to the original pH by electrolysis of barium salt solution.

4.4.2 Reagents Reagents shall be in accordance with 6.7.2 of JIS Z 2615.

4.4.3 Devices and Materials Devices and materials shall be in accordance with 5. of JIS Z 2615.

4.4.4 Apparatus The apparatus shall be in accordance with 6.7.3 of JIS Z 2615 ⁽⁴⁾.

4.4.5 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver Make the weighing-out amount of sample the most suitable amount (usually 0.5 to 1.0 g) to the using apparatus. Select the ignition improver most suitable ⁽¹⁾ from those indicated in 5. (13) of JIS Z 2615, add the most suitable amount to the weighing-out amount of sample and combustion apparatus and mix thoroughly or cover on the sample.

4.4.6 Operation

4.4.6.1 Preparatory Operation The preparatory operation shall be in accordance with 6.7.4 of JIS Z 2615.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1400 to 1450°C ⁽²⁾.

Further, in the case of using a high frequency induction heating furnace, set the conditions ⁽³⁾ relating to the high frequency induction heating furnace.

4.4.6.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The operation shall be in accordance with 6.7.5 of JIS Z 2615.

(2) In the Case of Using a High Frequency Induction Heating Furnace

- (a) Place a crucible containing the sample and ignition improver on a receiving stand, and close the combustion tube. After substituting the air in the tube by feeding-in oxygen at the designated flow rate, actuate the high frequency induction heating furnace and at the same time return the indicating value to zero.
- (b) When the indicating value of indicator increases gradually and a definite value is indicated, read out the indicated value and cut the switch of high frequency to take out the crucible.

4.4.7 Blank Test The blank test shall be carried out according to either one of the following:

- (1) In the Case of Using Tubular Electric Resistance Heating Furnace
The blank test shall be in accordance with 6.7.6 of JIS Z 2615.
- (2) In the Case of Using High Frequency Induction Heating Furnace
By using a crucible put-in with only ignition improver of the same amount as the amount added to the sample without putting-in the sample, carry out the operation of 4.4.6.2 (2).

Further, in the case of using such ignition improver, that the induction electric current is not generated by high frequency current, add 0.5 to 1.0 g of iron or the like of known carbon content and as lower as possible in carbon content to carry out, and correct the carbon amount contained in the added iron or the like to take it as the blank test value.

4.4.8 Calculation The calculation shall be in accordance with 6.7.7 of JIS Z 2615.

4.5 Combustion - Infrared Absorptiometric Method (Integral Method)

4.5.1 Summary Heat the sample in oxygen gas flow to oxidize sufficiently the carbon to make carbon dioxide, feed this together with oxygen into the infrared absorption cell, and measure the infrared absorption amount due to carbon dioxide.

4.5.2 Devices and Materials Devices and materials shall be in accordance with 5. and 6.9.2 of JIS Z 2615.

4.5.3 Apparatus The apparatus shall be in accordance with 6.9.3 of JIS Z 2615 (5).

Note (5) A tubular electric resistance heating furnace may be used.

4.5.4 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver Make the weighing-out amount the most suitable amount (usually 0.5 to 1.0 g) to the using apparatus. Select the ignition improver most suitable (1) from those indicated in 5. (13) of JIS Z 2615, add the most suitable amount to the weighing-out amount of sample and combustion apparatus and mix thoroughly or cover on the sample.

4.5.5 Operation

4.5.5.1 Preparatory Operation The preparatory operation shall be in accordance with 6.9.4 of JIS Z 2615.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1400 to 1450°C (2).

Further, in the case of using a high frequency induction heating furnace, set the conditions (3) relating to the high frequency induction heating furnace.

4.5.5.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace

- (a) Insert a boat containing the sample and ignition improver into the middle part of combustion tube, and feed oxygen at a designated flow rate to the apparatus to be used.
- (b) When the indicating value of indicator is increased gradually and a definite value is indicated, read out the indicating value.

(2) In the Case of Using a High Frequency Induction Heating Furnace The operation shall be in accordance with 6.9.5 of JIS Z 2615.

4.5.6 Blank Test The blank test shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The blank test shall be in accordance with 6.7.6 of JIS Z 2615.

(2) In the Case of Using a High Frequency Induction Heating Furnace The blank test shall be in accordance with 6.9.6 of JIS Z 2615.

4.5.7 Calculation The calculation shall be in accordance with 6.9.7 of JIS Z 2615.

4.6 Combustion - Infrared Absorptiometric Method (Circulation Method)

4.6.1 Summary Heat the sample in a circulating oxygen gas flow at a definite pressure in a definite volume, oxidize carbon to carbon dioxide and carbon monoxide, feed together with the excess oxygen into the infrared absorption detector of circulation loop, and measure respectively infrared absorption amounts of carbon dioxide and carbon monoxide.

4.6.2 Devices and Materials Devices and materials shall be in accordance with 5. and 6.9.2 of JIS Z 2615.

4.6.3 Apparatus The apparatus shall be in accordance with 5. and 6.10.2 of JIS Z 2615 ⁽⁵⁾.

4.6.4 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver Make the weighing-out amount the most suitable amount (usually 0.5 to 1.0 g) to the apparatus to be used. Select the ignition improver most suitable ⁽¹⁾ from those indicated in 5. (13) of JIS Z 2615, add the most suitable amount to the weighing out amount of sample and combustion apparatus mix thoroughly or cover on the sample.

4.6.5 Operation

4.6.5.1 Preparatory Operation The preparatory operation shall be in accordance with 6.10.3 of JIS Z 2615.