

JIS

JAPANESE INDUSTRIAL STANDARD

**Methods for Determination of
Hydrogen in Zirconium
and Zirconium Alloys**

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In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

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Methods for Determination of Hydrogen in
Zirconium and Zirconium Alloys

H 1664-1988

1. Scope

This Japanese Industrial Standard specifies the methods for determination of hydrogen in zirconium and zirconium alloys.

Remark: The units given in { } in this Standard are in accordance with the International System of Units (SI) and are appended for informative reference.

Further, the part where conventional units and units by SI given in { } thereafter are given in this Standard shall be converted to units specified in Appendix on and after January 1, 1991.

2. General Matters

General matters common to the methods of chemical analysis shall be in accordance with JIS H 1650 and JIS Z 2614.

3. Classification of Determination Methods

The method for determination of hydrogen shall be in accordance with either one of the following methods:

- (1) Vacuum Heating-Constant Volume Manometric Method This method applies to the sample of 0.0001 wt % and over to 0.005 wt % excl. in hydrogen content.
- (2) Inert Gas Fusion-Gas Chromatography This method applies to the sample of 0.0001 wt % and over to 0.005 wt % excl. in hydrogen content.

4. Preparation of Sample

The preparation of a sample shall be in accordance with either one of the following methods:

- (1) File Polishing The file polishing shall be as prescribed in 4.5.1 (1) of JIS Z 2614.
- (2) Chemical Polishing After dipping a sample into the mixed solution of 100 ml nitric acid (1 + 1) and 5 ml hydrofluoric acid, it is successively washed with water, ethanol, and acetone for each one minute by using an ultrasonic cleaner and dried by sending air.

Applicable Standards:

JIS H 1650-General Rules for Chemical Analysis of Zirconium and Zirconium Alloys

JIS Z 2614-General Rules for Determination of Hydrogen in Metallic Materials

5. Weighing-Out Amount of Sample

The weighing-out amount of a sample shall be 0.3 g.

6. Vacuum Heating-Constant Volume Manometric Method

6.1 Summary A sample is heated with an electric resistance heating furnace under vacuum and hydrogen together with other gases are extracted to be collected in a constant volume. Then, its pressure is measured. Hydrogen in this collected gas is oxidized by heated copper (II) oxide to prepare water. After absorbing it in diphosphorus pentoxide, the pressure is measured.

6.2 Materials and Reagents The materials and reagents shall be in accordance with 3.2.2 (1) of JIS Z 2614.

6.3 Apparatus For the apparatus, the gas extraction part of 3.2.2 (2) in JIS Z 2614⁽¹⁾⁽²⁾ is connected to the gas analysis part of 3.3.1 (2) in JIS Z 2614 to be used.

Notes ⁽¹⁾ For the heating apparatus, a tubular electric resistance heating furnace is used and a high frequency induction heating system is not used.

⁽²⁾ A furnace tube prepared by sealing one end of a transparent quartz tube of 25 to 40 mm in bore and 300 to 400 mm in length and fitting another end thereof to a water cooling type glass cap by grinding is used as the extraction furnace. The furnace tube is equipped with a side tube for connecting the furnace tube to a gas extraction pump and a side tube for filling a sample. An example of the extraction furnace is given in Attached Fig. 1.

6.4 Operation

6.4.1 Preparatory Operation The preparatory operation shall be carried out in accordance with the following procedures:

- (1) Connect a furnace tube to a water cooling type glass cap by using vacuum grease, insert the furnace tube into the heating part of a tubular electric resistance heating furnace and fix the heating furnace so that the bottom part of the furnace tube is positioned at the centre of the heating part.
- (2) Carry out the operation in accordance with the procedures of 5.1.3 (1) (b) to 5.1.3 (2) (d) of JIS Z 2614⁽³⁾.

Note ⁽³⁾ In the operation of 5.1.3 (2) (c) to (d) of JIS Z 2614, operate a high frequency induction oscillator and a blower, heat a graphite crucible at a degassing temperature and degas it for several hours. Thereafter, lower it to a gas extraction temperature. However, instead of this operation, apply power to a tubular electric resistance heating furnace and heat the furnace tube at 1100 to 1150°C in gas extraction temperature.

6.4.2 Determination Operation The determination operation shall be carried out in accordance with 5.1.4 (2) of JIS Z 2614. However, though a graphite crucible is heated at a gas extraction temperature in the operation of 5.1.4 (2) (a-1) of JIS Z 2614, a furnace tube is kept at a gas extraction temperature instead of it. The gas extraction temperature shall be 1100 to 1150°C and gas collection time shall be 15 minutes.

6.5 Blank Test The determination operation of 6.4.2 shall be carried out without using a sample.

6.6 Calculation The calculation shall be as prescribed in 5.1.4 (3) of JIS Z 2614. However, a conversion factor shall be preliminarily calculated in accordance with 5.1.4 (4) of JIS Z 2614.

7. Inert Gas Fusion-Gas Chromatography

7.1 Summary A sample together with tin is fused by heating with a graphite crucible according to an impulse system in a current of inert gas and hydrogen is extracted together with other gases. By passing the extracted gas as it is through a separation column, or otherwise by oxidizing the hydrogen in the extracted gas to be converted to water and then, passing through the separation column, hydrogen or water is separated from other gases, introduced to a heat conductivity detector and the variation of heat conductivity due to hydrogen or water is measured.

7.2 Materials and Reagents The materials and reagents shall be as follows:

- (1) Tin (Granular or Disklike)
- (2) Argon or Helium⁽⁴⁾ That of not less than 99.99 vol %.
- (3) Hydrogen That of not less than 99.99 vol %.
- (4) Graphite Crucible That conforming to an impulse furnace. Its example is given in Attached Fig. 2.

Note ⁽⁴⁾ Argon is used in the case where the extracted hydrogen is measured as it is and helium is used in the case where it is measured after conversion to water.

7.3 Apparatus The apparatus is composed of an inert gas cleaning part, a gas extraction part, a gas separation part, a gas measurement part, a hydrogen cleaning part for inspection, a gas inspection part, etc. (Attached Fig. 3 and Attached Fig. 4)⁽⁵⁾.

Note ⁽⁵⁾ The constitution, structure and use conditions of an apparatus is different according to the apparatus to be used.

- (1) Inert Gas Cleaning Part The inert gas cleaning part is composed of an oxidation tube, a carbon dioxide absorption tube, a dehydration tube, an electric resistance heating furnace, etc.
 - (a) Oxidation Tube A stainless steel tube packed by copper (II) oxide (granular). It is heated in the electric resistance heating furnace to be used.