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JAPANESE INDUSTRIAL STANDARD

Methods for Determination of Tungsten
in Iron and Steel

JIS G 1220-1980



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Translated

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Methods for Determination of Tungsten in
Iron and Steel

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1. Scope

This Japanese Industrial Standard specifies the methods for determination of tungsten in iron and steel.

2. General Matters The general matters in common with the determination shall be in accordance with JIS G 1201.

3. Classification of Methods The method for determination of tungsten in iron and steel shall be in accordance with one of the following:

- (1) Tungsten trioxide gravimetric method This method is applicable to the sample of 0.5 % or over in tungsten content.
- (2) Hydroquinone absorptiometry This method is applicable to the sample of 0.5 % and over, up to 7 % in tungsten content.

Applicable Standards:

JIS G 1201-General Rules for Chemical Analysis of Iron and Steel

JIS G 1217-Methods for Determination of Chromium in Iron and Steel

JIS G 1218-Methods for Determination of Molybdenum in Iron and Steel

JIS G 1221-Methods for Determination of Vanadium in Iron and Steel

JIS K 8006-Fundamental Articles on Volumetric Determinations

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- (3) Ammonium thiocyanate absorptiometry This method is applicable to the sample of 0.05 % and over, up to 7 % in tungsten content.
- (4) TPAC-potassium thiocyanate-chloroform extraction absorptiometry This method is applicable to the sample of 0.01 % and over, up to 7 % in tungsten content.

4. Tungsten Trioxide Gravimetric Method

4.1 Summary The sample is decomposed with suitable acid and tungsten is converted to tungstic acid, and the tungsten is allowed to deposit completely with cinchonine. The precipitate is filtered out, and after ignited this is treated with sulfuric acid and hydrofluoric acid to eliminate silicon dioxide, ignite again and mass of impure tungsten trioxide is weighed. Next, this is fused with sodium carbonate, dissolved with warm water and filtered; insoluble residue is ignited and its mass is weighed, and this is subtracted from the mass of the impure tungsten trioxide.

4.2 Field of Application In the case where the sample is difficult to be decomposed with hydrochloric acid and nitric acid, preparation of the sample solution shall be carried out in separated operation, and in the case where niobium and tantalum are contained, separation of the tungsten trioxide shall be carried out in other operation.

Furthermore, in the case where chromium, molybdenum, vanadium, and others are contained, correction shall be made with determining these elements in the impure tungsten trioxide.

4.3 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1+1)
- (3) Nitric acid

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- (4) Perchloric acid
- (5) Hydrofluoric acid
- (6) Hydrofluoric acid (1+10)
- (7) Sulfuric acid (1+1)
- (8) Phosphoric acid
- (9) Aqueous ammonia
- (10) Sodium hydroxide solution (50 w/v %)
- (11) Sodium carbonate (anhydride)
- (12) Potassium carbonate (anhydride)
- (13) Iron solution (10 mg Fe/ml) The solution prepared as follows: Heat to decompose 1.000 g of iron, which is higher in purity as far as possible and does not contain molybdenum or of which molybdenum content is smaller as far as possible and of known content, with 10 ml of perchloric acid, and cool after white fume treated, transfer into a measuring flask of 100 ml, and dilute up to the marked line with water.
- (14) Magnesium sulfate-ammonium chloride solution The solution prepared as follows: Weigh out 10 g of magnesium sulfate and 20 g of ammonium chloride and transfer into a beaker (300 ml), add 2 ml of aqueous ammonia, and after diluted to 250 ml with water heat this quietly while mixing with stirring to dissolve, and cool. This solution shall be prepared on each occasion of use.

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- (15) Magnesium sulfate washing solution The solution prepared as follows: Weigh out 10 g of magnesium sulfate and 20 g of ammonium chloride, transfer into a beaker (1000 ml), add an adequate amount of water and 4 ml of aqueous ammonia, and heat quietly to dissolve. Further, add 10 g of potassium carbonate and heat quietly while mixing with stirring, to dissolve, and after cooled to ordinary temperature dilute this to 1000 ml with water. This solution shall be prepared on each occasion of use.
- (16) Tartaric acid solution (50 w/v %)
- (17) Cinchonine solution The solution prepared by dissolving 12.5 g of cinchonine ($C_{11}H_{11}N_2O$) in 100 ml of the hydrochloric acid (1+1).
- (18) Cinchonine washing solution The solution prepared by diluting 30 ml of the cinchonine solution [4.3 (17)] in 1000 ml of water.
- (19) Rhodamine B solution (1 w/v %) The solution prepared by dissolving 2 g of Rhodamine B ($C_{21}H_{19}O_2N_2Cl$) in 200 ml of warm water. This solution shall be prepared on each occasion of use.
- (20) Rhodamine B washing solution The solution prepared by diluting 10 ml of Rhodamine B solution [4.3 (19)] to 1000 ml with water, and adding 3 ml of the hydrochloric acid to this. This solution shall be prepared on each occasion of use.
- (21) Methyl Orange indicator (0.1 w/v %) The method of preparation shall be in accordance with 3. of JIS K 8006.

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4.4 Mass of Sample to Be Weighed-Out The sample shall generally be weighed-out in accordance with Table 1.

Table 1

Tungsten content %	Mass of sample to be weighed-out g
0.5 and over, up to 1	5.0
1 and over, up to 10	2.0
10 and over	1.0

4.5 Operations

4.5.1 Preparation of Sample Solution The preparation of the sample solution shall be carried out as follows:

- (1) Sample Easily to Be Decomposed with Hydrochloric Acid and Nitric Acid Transfer the weighed-out sample into a beaker (300 ml), cover with a watch glass, add 50 to 80 ml of hydrochloric acid (1+1) corresponding to the weighed-out mass of the sample, and heat to decompose. Add to this 5 to 10 ml of the nitric acid, and boil to convert tungsten to tungstic acid. Remove the watch glass, evaporate until liquid quantity becomes approximately 15 ml, cover again with the watch glass, further heat to generate nitrogen oxide and the like, condense to approximately 10 ml (¹), and cool. Next, add 10 ml of hydrochloric acid (1+1) and warm water to make the liquid quantity approximately 100 ml.
- (2) Sample Difficult to Be Decomposed with Hydrochloric Acid and Nitric Acid Transfer the weighed-out sample into a beaker (300 ml), cover with a watch glass, add respectively 20 to 30 ml (²) of the hydrochloric acid and the nitric acid, and heat to decompose. Next, add 15 ml of perchloric acid, and after added with 7 ml per 1 g of the sample, add 1 to 3 ml

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of the hydrofluoric acid and continue heating, oxidize chromium allowing to generate white fume of the perchloric acid, and succeedingly allow to generate white fume for about 5 min. After cooling, add warm water to make the liquid quantity approximately 200 ml.

Notes ⁽¹⁾ In the case of the sample of 5 % or over in tungsten content such as tool steel and high speed steel, add 5 ml of hydrofluoric acid (1+10), and heat succeedingly to condense the liquid quantity to approximately 5 ml.

⁽²⁾ The quantities of hydrochloric acid and nitric acid used shall be altered appropriately according to the type, shape, weighed-out mass, and others of the samples.

4.5.2 Separation and Weighing of Tungsten Trioxide

(1) In the Case Where Niobium and Tantalum Are Not Contained

(1.1) Add 5 ml of the cinchonine solution [4.3 (17)] to the sample solution obtained in 4.5.1 and heat, hold for 30 to 60 min at about 90°C while mixing with shaking from time to time. In the case of a smaller amount of tungsten, leave alone further for one night at room temperature. After cooling, filter out by using filter paper (Class 5 C or Class 6) added with a small amount of paper pulp, and wash with the warm cinchonine washing solution [4.3 (18)] until iron ion in the washings disappears. Discard the filtrate and washings. Transfer the precipitate together with the filter paper pulp, as wetted, into a platinum crucible (No. 30) of known mass. In the case where any tungstic acid is adhering to the inner wall of the beaker, wipe off with a small piece of filter paper (Class 6) wetted with aqueous ammonia, and combine with the main precipitate contained in the platinum crucible.

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Heat quietly to dry, increase temperature gradually, and after the filter paper and others have been incinerated ignite within a range of 750 to 800°C, and cool. Add 2 to 3 drops of sulfuric acid (1+1) and 3 to 5 ml of the hydrofluoric acid to this, heat and evaporate to dryness.

- (1.2) Ignite the platinum crucible containing the precipitate at a range of 750 to 800°C, cool to ordinary temperature in a desiccator and weigh the mass. Take this as the amount of impure tungsten trioxide.

(2) In the Case Where Niobium and Tantalum Are Contained

- (2.1) Operate in accordance with the procedures of (1.1) of 4.5.2 (1).

- (2.2) Ignite the platinum crucible containing the precipitate at a range of 750 to 800°C and cool. Add 4 g of the potassium carbonate (anhydride) to this and heat to fuse, after cooling, add approximately 20 ml of warm water, heat slowly and dissolve melts. After this solution has been transferred into a beaker (500 ml) add water to make the liquid quantity approximately 200 ml, add 25 ml of magnesium sulfate-ammonium chloride solution [4.3 (14)] and mix with shaking well, and leave alone for 1 to 4 h. Filter the precipitate through filter paper (Class 6). Wash 5 to 6 times with the magnesium sulfate washing solution [4.3 (15)], and discard the filter paper and residue. Collect the solution and washings into a beaker (500 ml), add 1 to 2 drops of Methyl Orange indicator [4.3 (21)], and after this has been dropped with the hydrochloric acid until the colour of the solution turns to red from yellow, add 1 ml of the hydrochloric acid per 100 ml of the solution. After this solution has been heated to boil, add 20 ml of Rhodamine B solution [4.3 (19)] while mixing with stirring, boil for 1 to 2 min and allow the precipitate to aggregate. Leave standing for a