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JAPANESE INDUSTRIAL STANDARD

Methods for Determination of Molybdenum
in Iron and Steel

JIS G 1218-1980



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Methods for Determination of

G 1218-1980

Molybdenum in Iron and Steel

1. Scope

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

2. General Matters

The general matters in common with methods for determination shall be in accordance with JIS G 1201.

3. Classification of Methods

The methods for determination of molybdenum in iron and steel shall be in accordance with one of the following:

- (1) Method of determination by α -benzoinoxime separation This method applies to a sample not less than 0.03 % in molybdenum content.
- (a) Gravimetric method
- (b) Absorptiometry

Applicable Standards:

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

JIS K 8006-Fundamental Articles on Volumetric Determinations

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- (2) Sodium thiocyanate absorptiometry This method applies to a sample less than 9 % in molybdenum content.
- (3) Sodium thiocyanate extraction absorptiometry This method applies to a sample less than 0.03 % in molybdenum content.

4. Method of Determination by α -Benzoinoxime Separation

4.1 Summary The sample is decomposed in a suitable acid, and after iron and others have been oxidized, it is filtered. Ferrous ammonium sulfate is added to the filtrate to reduce vanadium and others, and molybdenum has been separated from iron and others by precipitating with α -benzoinoxime, and then the gravimetric method or absorptiometry is applied.

- (1) Gravimetric Method After the precipitate has been dried, it is incinerated to make impure molybdenum oxide, and its mass is weighed. Next, the impure molybdenum oxide is dissolved with aqueous ammonia, insoluble matter is filtered off, dried, incinerated, and its mass is weighed, and this is subtracted from the mass of the impure molybdenum oxide.
- (2) Absorptiometry The precipitate is decomposed with nitric acid, sulfuric acid, phosphoric acid, and perchloric acid, and after white-fume treated, sulfuric acid, sodium thiocyanate, sodium iodide, and ascorbic acid are allowed to coexist, and absorbancy of molybdenum thiocyanate complex which is generated is measured.

4.2 Field of Application In the case where the sample contains tungsten, in the case where it is difficult to decompose with sulfuric acid and nitric acid, in the case where niobium is contained, and in the case where it is difficult to decompose with acid, preparation of respective sample solutions shall be made by separate operations.

Furthermore, in the case where a large amount of tungsten is contained, after filtered off as tungstic acid, an amount of molybdenum which has been adsorbed in the precipitate is corrected.

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4.3 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1+1, 2+100)
- (2) Nitric acid
- (3) Perchloric acid
- (4) Sulfuric acid
- (5) Sulfuric acid (1+1, 3+7, 1+5, 2+100)
- (6) Sulfurous acid water (saturated)
- (7) Phosphoric acid
- (8) Aqua regia (hydrochloric acid 3, nitric acid 1)
- (9) Aqueous ammonia
- (10) Sodium hydroxide solution (50 w/v %)
- (11) Iron Iron which is as higher as possible in purity, does not contain molybdenum or is as smaller as possible in molybdenum content, and also the content is known.
- (12) Bromine water (saturated)
- (13) Fusing mixture [sodium carbonate (anhydrous) 1, potassium carbonate (anhydrous) 1]
- (14) Iron solution: That has been prepared as follows: Weigh out 0.500 g of iron [4.3 (11)], transfer into a beaker (200 ml), cover with a watch glass, add 20 ml of the perchloric acid to decompose by heating, and concentrate by heating succeedingly to generate white fume of the perchloric acid. After cooling to ordinary temperature, add 70 ml of the perchloric acid to dissolve salts, transfer into a measuring flask of 100 ml, and dilute up to the marked line with water.
- (15) Potassium iodide solution (30 w/v %)
- (16) Ammonium thiocyanate solution (20 w/v %)
- (17) Sodium thiocyanate solution (10 w/v %)

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- (18) Stannous chloride solution that has been prepared as follows: dissolve by heating 50 g of stannous chloride (di-hydrate) in 200 ml of hydrochloric acid (1+1), and after cooled to ordinary temperature, dilute to 500 ml with water. Store this solution in a brown bottle with adding approx. 2 g of granular tin.
- (19) Ammonium ferrous sulfate solution that has been prepared as follows: after 100 g of ammonium ferrous sulfate (6-hydrate) has been dissolved in an adequate amount of water and 100 ml of the sulfuric acid (1+1), dilute to 1000 ml with water.
- (20) Tartaric acid solution (50 w/v %)
- (21) L-ascorbic acid solution (1 w/v %) Prepare this solution at each time of use.
- (22) α -benzoinoxime solution that has been prepared as follows: 10 g of α -benzoinoxime ($C_{11}H_{13}NO_2$) has been dissolved into 500 ml of methyl alcohol.
- (23) α -benzoinoxime cleansing solution that has been prepared as follows: mix approx. 40 ml of the α -benzoinoxime solution [4.3 (22)] with 1000 ml of sulfuric acid (1+99). Prepare this solution, at each time of use.
- (24) Standard molybdenum solution (100 μ g Mo/ml) that has been prepared as follows: weigh out 1.8402 g of ammonium molybdate (tetrahydrate), and after it has been dissolved into warm water, cool to ordinary temperature, transfer into a measuring flask of 1000 ml, and dilute up to the marked line with water to take this as the standard stock solution (1 mg Mo/ml). At each time of use, separately take a definite volume of this solution and dilute to 10 times with water correctly to prepare for the standard molybdenum solution. Determine the concentration of the standard stock solution by the α -benzoinoxime separation gravimetric method.

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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

Gravimetric method		Absorptiometry	
Molybdenum content %	Mass of sample to be weighed out g	Molybdenum content %	Mass of sample to be weighed out g
0.03 and over, up to 1 excl.	2.0	0.03 and over, up to 0.5 excl.	2.0
1 and over, up to 3 excl.	1.0	0.5 and over, up to 1 excl.	1.0
3 min.	0.50	1 and over, up to 2 excl.	0.50
		2 and over, up to 5 excl.	0.20
		5 min.	0.10

4.5 Operations

4.5.1 Preparation of Sample Solutions The preparation of sample solutions shall be in accordance with the following:

(1) Sample Easy to Decompose with Sulfuric Acid and Nitric Acid

- (1.1) Transfer the weighed out sample to a beaker (300 ml), cover with a watch glass, add 40 ml of the sulfuric acid (1+5) and heat to decompose. Add 5 ml of nitric acid to this to boil, and oxidize iron, molybdenum and others. Heat succeedingly to concentrate until white fume of the sulfuric acid is generated.
- (1.2) After cooled for a while, add approx. 50 ml of water, boil to dissolve salts, filter using filter paper (Class 5 B), and rinse thoroughly with warm sulfuric acid (2+100). Collect the filtrate and washings into a beaker (300 ml), and dilute to approx. 100 ml with water. After having cooled to about 25°C, add 5 to 10 ml of the ammonium ferrous sulfate solution to reduce vanadium.

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- (2) Sample Containing Tungsten Transfer the weighed out sample into a beaker (300 ml), cover with a watch glass, add 20 to 50 ml of the hydrochloric acid (1+1) corresponding to the weighed out mass of the sample, and heat to decompose. Add 5 ml of nitric acid to this, boil to oxidize the tungsten, heat to evaporate successively to concentrate until a coating is generated.

Next, add 10 ml of hydrochloric acid (1+1) and heat to dissolve, add approx. 50 ml of warm water and after having boiled for 1 to 2 min, hold it for about 1 h at 90°C. Filter tungstic acid using filter paper (Class 5 B) or filter paper pulp, and rinse thoroughly with warm hydrochloric acid (2+100) ⁽¹⁾. Collect the filtrate and washings into a beaker (300 ml), add 10 ml of the sulfuric acid (1+1) and heat to concentrate until white fume of the sulfuric acid is generated. Hereinafter, operate in accordance with the procedures of 4.5.1 (1.2).

- (3) Sample Difficult to Decompose with Sulfuric Acid and Nitric Acid and Sample Containing Niobium Transfer the weighed out sample into a beaker (300 ml), cover with a watch glass, add 20 to 50 ml of the aqua regia correspondingly to the weighed out mass of sample and heat to decompose. Add 30 ml of perchloric acid to this and heat to generate dense white fume of the perchloric acid and oxidize chromium to dichromate. After having cooled for a while, add approx. 100 ml of warm water to dissolve salts, add 30 ml of the sulfurous acid water (saturated), and boil to reduce chromium. Filter using filter paper (Class 5 B), and rinse thoroughly with warm sulfuric acid (2+100). Collect the filtrate and washings into a beaker (300 ml) and discard the residue.

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- (4) Sample Difficult to Decompose with Acid Transfer the weighed out sample into a beaker (300 ml), cover with a watch glass, add 40 ml of the sulfuric acid (1+5), heat to decompose, add 5 ml of nitric acid and boil to oxidize iron and others. After leaving cooled for a while, filter using filter paper (Class 5 B), and rinse thoroughly with warm sulfuric acid (2+100). Collect the filtrate and washings into a beaker (300 ml), and store as the principal solution.

Transfer the residue together with the filter paper into a platinum crucible (No. 30), and incinerate the filter paper at 500°C or under.

Mix this with about 10 times in volume of the fusing mixture [4.3 (13)] and heat to melt. After cooling, dissolve the melt with a small quantity of hydrochloric acid (1+1) and water, and combine with the principal solution. Heat this to concentrate until white fume of the sulfuric acid is generated. Hereafter, operate in accordance with the procedures of 4.5.1 (1.2).

Note ⁽¹⁾ Molybdenum which has been adsorbed to the tungstic acid being filtered off shall be operated in accordance with the procedures of 4.5.3 (2), and be corrected by otherwise carrying out determination.

4.5.2 Precipitation Forming of Molybdenum Cool the sample solution obtained in 4.5.1 to 10°C or under ⁽²⁾, add 10 ml of the α -benzoinoxime solution gradually while mixing with stirring, and further add in excess 5 ml of the α -benzoinoxime solution per 10 mg of molybdenum content. Add the bromine water (saturated) until the solution presents yellow colour while mixing with stirring the solution ⁽³⁾, and further add a small amount of α -benzoinoxime solution ⁽⁴⁾.

Mix with stirring at times while cooling, after leaving for 10 min, filter out using filter paper (Class 5 A) put with a small quantity of filter paper pulp in it. Rinse the precipitates thoroughly with the α -benzoinoxime rinsing solution. Discard the filtrate and washings.