

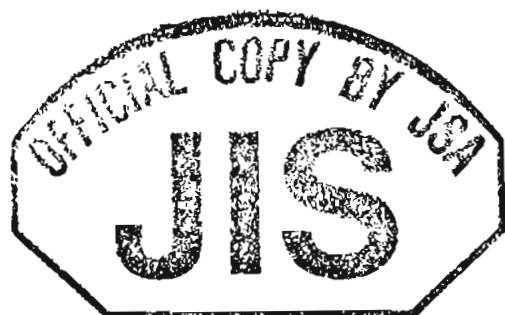
JIS

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JAPANESE INDUSTRIAL STANDARD

Methods for Determination of Phosphorus in Iron Ores

JIS M 8216—1983



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Methods for Determination of
Phosphorus in Iron Ores

M 8216-1983

1. Scope

This Japanese Industrial Standard specifies the methods for determination of phosphorus in iron ores.

2. General Matters

General matters common to the chemical analysis of iron ores shall be as prescribed in JIS M 8202.

3. Division of Methods

The methods for determination of phosphorus shall conform to any of the following methods:

- (1) Molybdenum Blue Absorptiometry This method shall be applied to the sample of under 0.5 % in phosphorus content.
- (2) Bismuth Addition Molybdenum Blue Absorptiometry This method shall be applied to the sample of under 0.5 % in phosphorus content.
- (3) Neutralization Titrimetric Method This method shall be applied to the sample of 0.01 % or over in phosphorus content.

Applicable Standards:

JIS K 8006-Fundamental Articles on volumetric Determinations

JIS M 8202-General Rules for Chemical Analysis of Iron Ores

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4. Molybdenum Blue Absorptiometry

4.1 Summary Decompose a sample with hydrochloric acid, nitric acid and perchloric acid, filter the insoluble residue and reserve the filtrate as a main solution. After treating the insoluble residue with hydrofluoric acid, fuse it with potassium disulfate and dissolve the melt with hydrochloric acid. Thereafter, collect the phosphorus as iron phosphate and join it to the main solution. Otherwise, fuse the sample with sodium peroxide, dissolve the melt with warm water, dissolve it in sulfuric acid and hydrogen peroxide and prepare the sample solution. Reducing iron or the like with sodium hydrogensulfite, measure the absorbance on phosphorus changed into molybdenum blue by ammonium molybdate and hydrazinium sulfate (2^+).

4.2 Applied Field When a sample contains 1 % or over of titanium dioxide, the fusion method can not be applied to the preparation of sample solution.

4.3 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1 + 1, 10 + 6)
- (3) Nitric acid
- (4) Perchloric acid
- (5) Hydrofluoric acid
- (6) Hydrobromic acid
- (7) Sulfuric acid (1 + 1)
- (8) Aqueous ammonia
- (9) Hydrogen peroxide water (1 + 9)
- (10) Sodium peroxide
- (11) Sodium hydrogensulfite solution (10 w/v %)
- (12) Potassium disulfate (potassium pyrosulfate)
- (13) Hydrazinium sulfate (2^+) (hydrazine sulfate) sulfuric acid solution
Add 15 ml sulfuric acid (1 + 1) and 75 ml water to 10 ml hydrazinium sulfate (2^+) solution (0.15 w/v %) and mix them by shaking.

- (14) Colouration reagent solution Mix 25 ml ammonium molybdate solution [dissolve 20 g ammonium molybdate (4 hydrate) $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$] with 100 ml warm water, add 600 ml sulfuric acid (1 + 1), cool it and dilute it to 1000 ml], 10 ml hydrazinium sulfate (2^+) solution (0.15 w/v %) and 65 ml water for each service.
- (15) Iron (III) chloride solution Weigh out 0.300 g high pure iron, transfer it into a beaker (300 ml), cover it with a watch glass, add 10 ml hydrochloric acid and quietly decompose it by heating. After oxidizing iron by adding 3 ml nitric acid, add 5 ml perchloric acid, evaporate it by heating and sufficiently generate white fume. After cooling, transfer it into a separatory funnel (100 ml) by washing with 20 ml hydrochloric acid (10 + 6). Add 20 ml 4-methyl-2-pentanone (MIBK), violently mix them by shaking for about one min and let it stand still. Thereafter, discard the lower layer, add 20 ml water to the separatory funnel and violently mix them by shaking for about one min. After standing still, transfer the lower layer to a beaker (300 ml). Add 5 ml water to the separatory funnel and collect the lower layer in the same beaker in the same way. After boiling it for a while, add 5 ml nitric acid and 10 ml perchloric acid and evaporate them by heating. When thick white fume starts to be generated, cool it, add 10 ml hydrochloric acid, dissolve it by heating and oxidize it by adding several drops nitric acid. After boiling, cool it to ordinary temperature, transfer it into a measuring flask of 100 ml and dilute it to the marked line with water.
- (16) Standard phosphorus solution A (100 $\mu\text{gP/ml}$) Dry potassium dihydrogenphosphate (KH_2PO_4) at 105°C to constant weight and cool it in a desiccator to ordinary temperature. Weigh out 0.4393 g said potassium dihydrogenphosphate, dissolve it with water, transfer it into a measuring flask of 1000 ml and dilute it to the marked line with water.
- (17) Standard phosphorus solution B (10 $\mu\text{gP/ml}$) Accurately dilute standard phosphorus solution A 10 times with water for each service.

4.4 Weighing-Out Amount of Sample A sample shall be, as a rule, weighed out in accordance with Table 1.

Table 1

Phosphorus content %	Weighing-out amount of sample g	
	Acid decomposition method	Alkali fusion method
Under 0.2	0.50	1.0
0.2 to 0.5 excl.	0.50	0.50

4.5 Operation

4.5.1 Preparation of Sample Solution The sample solution shall be prepared as follows.

(1) Acid Decomposition Method

- (1.1) Transfer the weighed-out sample into a beaker (300 ml), cover it with a watch glass, add 20 ml hydrochloric acid, at first place the beaker in the low temperature region (60 to 100°C) in the vicinity of a hot plate and keep it as it is for about one hour. Thereafter, transfer it to the high temperature region and decompose it by heating for about 10 min to near boiling. Then, add 5 ml nitric acid and 15 ml perchloric acid and continue to evaporate it by heating. Heat it for about 10 min in a state in which white fume starts to be generated in the beaker, its inside becomes transparent and the vapour of perchloric acid is refluxed along the inside walls of beaker. After cooling, dissolve the soluble salts by adding about 50 ml warm water, filter the insoluble residue by using a filter paper (Class 5 B) and filter paper pulp and sufficiently wash it with warm water. Collect the filtrate and washing in a beaker (300 ml) and preserve it as a main solution.
- (1.2) Transfer the insoluble residue together with the filter paper into a platinum crucible (No. 30). After drying, incinerate it by ignition. thereafter, cool it. Moisten the residue on ignition with sulfuric acid (1 + 1), add about 5 ml hydrofluoric acid and vapourize silicon dioxide and sulfuric acid by quietly heating them. After cooling, add about 3 g potassium disulfate, cover it, at first slowly heat it, gradually raise temperature and fuse the residue by heating it to a dark red heat state. After cooling, put the platinum crucible into a beaker (300 ml) and dissolve it by adding about 30 ml warm water and about 5 ml hydrochloric acid. Wash the platinum crucible with water and take it out. Accurately add 10 ml iron (III) chloride solution [4.3 (15)] and add little by little aqueous ammonia mixing them by shaking. When the generation of precipitate is completed and the solution is slightly alkalized, stop the addition. After boiling for about 2 min, take it down from the heat source, let it stand still and settle the precipitates of iron phosphate, iron hydroxide, etc. Filter those precipitates by using a filter paper (Class 5 A) and wash them with warm water. Discard the filtrate and washings at this time. Dissolve the precipitate by pouring about 10 ml warm hydrochloric acid (1 + 1) from above the filter paper and join it to the main solution reserved in (1.1). Concentrate this solution ⁽¹⁾ by heating and continue to heat it for about three min under a state in which thick white fume is refluxed along the inside walls of beaker. After cooling, dissolve the salts by adding about 50 ml warm water. If any insoluble residue is observed at this time, filter it by using a filter paper (Class 5 B) and sufficiently wash it with warm water. Discard the insoluble residue then. Collect the filtrate and washings into a measuring flask of 250 ml and cool it to ordinary temperature. Thereafter, dilute it to the marked line with water.

Note (¹) When 0.10 mg or more arsenic is contained in the sample solution partially taken in accordance with Table 2, first add 5 ml hydrobromic acid to this solution and then concentrate it by heating.

- (2) Alkali Fusion Method Transfer the weighed-out sample into a nickel crucible (30 ml), add 5 g sodium peroxide and sufficiently mix them with a platinum wire. Cover it and at first slowly heat it at a low temperature. After the mixture reaches a sintered state, gradually raise temperature. After the mixture is fused in a dark red heat state, take it down from the heat source and let it stand to cool. Put the cooled nickel crucible into the beaker (300 ml) containing 100 ml warm water, dissolve the melt, add 20 ml sulfuric acid (1 + 1) and quietly heat it. Thereafter, dissolve the oxides or the like by adding 1 ml hydrogen peroxide water (1 + 9). Wash the nickel crucible with water and take it out (²). Decompose excess hydrogen peroxide by boiling the solution for 3 to 5 min. After cooling to ordinary temperature, filter it by using a filter paper (Class 5 A) and sufficiently wash it with water. Collect the filtrate and washings into a measuring flask of 250 ml and dilute it to the marked line with water.

Note (²) If the inside wall of crucible is rubbed with a glass rod or the like when the crucible is taken out, black nickel oxide tends to be exfoliated and mixed, in the solution. Therefore, the nickel crucible shall be treated with only washing by spraying water.

4.5.2 Colouration Partially take the sample solution obtained in 4.5.1 in accordance with Table 2 according to the content of phosphorus and transfer it into a measuring flask (³) of 100 ml. Add 10 ml sodium hydrogensulfite solution (10 w/v %) to each flask, heat it in a boiling water bath and reduce iron (⁴) until the colour of solution stops changing. Add 25 ml colouration reagent solution [4.3 (14)] and continue to heat it again in a boiling water bath for ten min. After cooling it to ordinary temperature, dilute it to the marked line with water.

Table 2

Phosphorus content %	Sample solution aliquot amount ml	
	Acid decomposition method	Alkali fusion method
Under 0.2	25	10
0.2 to 0.5 excl.	10	10