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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently, **JIS G 1214:1992** is replaced with **JIS G 1214:1998**.

In this revision, **ISO 10714** has been employed in Annex 2 for conforming to International Standard.

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Iron and steel — Methods for determination of phosphorus content

Introduction This Japanese Industrial Standard is prepared by citing the translation of **ISO 10714**, *Steel and iron — Determination of phosphorus content — Phosphovanadomolybdate spectrometric method* issued in 1992 in Annex 2 without changing technical contents and the style. Items which are not specified in the corresponding International Standard are added as JIS.

The underlined “informative references” in this Standard are items which do not exist in the original International Standard.

1 Scope This Japanese Industrial Standard specifies the methods for determination of phosphorus in iron and steel.

Remarks : The corresponding International Standard to this Standard is as follows :

ISO 10714 *Steel and iron – Determination of phosphorus content – Phosphovanadomolybdate spectrophotometric method*

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards indicated below shall be applied.

JIS G 1201 *General rules for chemical analysis of iron and steel*

JIS Z 8402 *General rules for permissible tolerance of chemical analyses and physical tests*

3 General matters General matters common to methods for determination shall be in accordance with **JIS G 1201**. However, **JIS G 1201** shall not apply to Annex 2.

4 Classification of methods for determination The methods for determination of phosphorus in iron and steel shall be in accordance with any one of the following :

- a) **Reduced molybdophosphate spectrophotometric method** This method shall apply to the sample of 0.005 % (m/m) or over up to and including 0.50 % (m/m) phosphorus content as specified in Annex 1 (normative).
- b) **Phosphovanadomolybdate spectrophotometric method (ISO 10714)** This method shall apply to the sample of 0.001 0 % (m/m) or over up to and including 1.0 % (m/m) phosphorus content, as specified in Annex 2 (normative).
- c) **Molybdophosphate extraction spectrophotometric method** This method shall apply to the sample of 0.000 3 % (m/m) or over up to and including 0.010 % (m/m) as specified in Annex 3 (normative). However, it can not apply to the sample containing 0.1 % (m/m) or over tungsten.
- d) **Reduced molybdophosphate spectrophotometric method after extraction of molybdophosphate** This method shall apply to the sample of 0.000 3 % (m/m) or over up to and including 0.010 % (m/m) phosphorus content as specified in Annex 4 (normative). However, it can not apply to the sample containing 0.1 % (m/m) or over tungsten.

Annex 1 (normative) Reduced molybdophosphate spectrophotometric method

1 Summary Decompose a sample with appropriate acid, add perchloric acid, and generate white fumes of perchloric acid by heating. After reducing iron or the like by adding sodium hydrogensulfite, generate molybdophosphate blue by adding hexaammonium heptamolybdate and hydrazinium sulfate, and measure the absorbance of the generated molybdophosphate blue with a photometer.

2 Reagents The reagents shall be as follows.

- a) **Hydrochloric acid**
- b) **Nitric acid**
- c) **Nitric acid (1+1)**
- d) **Perchloric acid**
- e) **Hydrofluoric acid (1+4)**
- f) **Boric acid**
- g) **Mixed acid** (1 part hydrochloric acid and 1 part nitric acid)
- h) **Aqueous ammonia**
- i) **Iron** It is the iron having as high purity as possible, which contains no phosphorus or of which the phosphorus content is as low as possible and known.
- j) **Beryllium sulfate solution** After dissolving 19.7 g beryllium sulfate tetrahydrate in 20 ml of sulfuric acid (1 + 1), dilute the solution with water to make up the volume to 1 000 ml.
- k) **Sodium hydrogensulfite solution** (100 g/l)
- l) **Hydrazinium sulfate sulfuric acid solution** Add 15 ml of sulfuric acid (7 + 5) and 75 ml of water to 10 ml of hydrazinium sulfate solution (1.5 g/l), and mix by shaking.
- m) **Disodium ethylenediaminetetraacetate dihydrate** (hereafter referred to as EDTA2Na)
- n) **EDTA2Na solution** After dissolving 3.0 g of EDTA2Na and 3.0 g of ammonium nitrate in 6 ml of aqueous ammonia, dilute the solution with water to make up the volume to 500 ml.
- o) **Color reagent solution** After mixing 25 ml of hexaammonium heptamolybdate solution and 10 ml of hydrazinium sulfate solution (1.5 g/l), dilute the solution with water to make up the volume to 100 ml, and mix sufficiently by shaking. Prepare this solution at each service.

Prepare the hexaammonium heptamolybdate solution as follows. Dissolve 20 g of hexaammonium heptamolybdate tetrahydrate in 600 ml of water, add 350 ml of sulfuric acid by stirring while the solution is cooled in running water, cool it to room temperature, and dilute the solution with water to make up the solution to 1 000 ml.

- p) **Standard phosphorus solution** (40 µgP/ml) Weigh 0.8787 g potassium dihydrogenphosphate which was previously made at 110 °C to constant mass and allowed to cool to ordinary temperature in a desiccator, transfer into a beaker (300 ml), dissolve by adding about 1 000 ml of water, transfer the solution into a 1 000 ml one mark volumetric flask by using water, and dilute to the mark with water to make stock solution (200 µgP/ml). Dilute only a necessary amount of this stock solution accurately five times with water at each service to make standard phosphorus solution.

3 Test portion The test portion of a sample shall be as given in Annex 1 Table 1.

Annex 1 Table 1 Weighing amount of sample

Phosphorus content % (m/m)	Test portion g
0.005 or over to and excl. 0.040	1.0
0.040 or over to and excl. 0.15	0.50
0.15 or over up to and incl. 0.50	0.15

4 Operation

Informative reference : **WARNING** The vapour of perchloric acid has a risk of explosion under existence of ammonia, nitrous acid vapour or organic matter. The treatment of perchloric acid vapour shall be carried out at a place equipped with an exhaust facility which ensures safety even though perchloric acid is used.

4.1 Preparation of sample solution Prepare a sample solution in accordance with any one of the following procedures.

a) Sample easily decomposed with nitric acid

- 1) Weigh a sample, transfer to a beaker (200 ml).
- 2) Cover with a watch glass, add 10 ml of nitric acid (1+1), decompose by heating, add 5 ml of perchloric acid for 0.15 g of the weighed sample, 10 ml for 0.50 g and 15 ml for 1.0 g, continue to heat, and keep such state for 5 min to 10 min, that the inside of the beaker becomes transparent and the vapour of perchloric acid is refluxed along the inner wall of the beaker ⁽¹⁾.

Note ⁽¹⁾ When the concentration of perchloric acid in coloration exceeds 0.9 mol/l, the coloration of phosphorus is remarkably disturbed. Therefore, treatment for generating white fumes shall be thoroughly performed.

- 3) After allowing to cool, wash the bottom surface of a watch glass with a small amount of water, remove the watch glass, add about 40 ml of water, dissolve salts by shaking, filter the solution into a one mark volumetric flask by use of filter paper (class 5A), wash several times with warm water, and combine the washings into filtrate. After cooling the solution to ordinary temperature, dilute to the mark with water. Discard the residue.

b) Pig iron and sample not easily decomposed with nitric acid

- 1) Weigh a sample, transfer to a beaker (200 ml).
- 2) Cover with a watch glass, add 15 ml of mixed acid, decompose by heating, add 5 ml of perchloric acid for 0.15 g of the weighed sample, 10 ml for 0.50 g and 15 ml for 1.0 g, continue to heat, and keep such state for 5 min to 10 min that the inside of the beaker becomes transparent and the vapour of perchloric acid is refluxed along the inner wall of the beaker ⁽¹⁾.
- 3) Carry out the operation stated in a) 3).

c) Sample containing 5 % (m/m) or over chromium

- 1) Weigh a sample, transfer to a beaker (200 ml).
- 2) Cover with a watch glass, add 15 ml of mixed acid, decompose by heating, add 10 ml of perchloric acid for 0.15 g of the weighed sample, 15 ml for 0.50 g and 20 ml for 1.0 g, and continue to heat. After oxidizing chromium to dichromate to the extent that the inside of the beaker becomes transparent and the vapour of perchloric acid is refluxed along the inner wall of the beaker, drip hydrochloric acid several times little by little to volatilize most of chromium as dioxochromium dichloride. After repeating the dripping of hydrochloric acid until brown fume is not generated, successively heat to generate white fume of perchloric acid ⁽¹⁾ until residual chromium oxidizes to dichromate.
- 3) Carry out the operation stated in a) 3)

d) Sample containing 0.1 % (m/m) or over tungsten

- 1) Weigh a sample, and transfer into a beaker (200 ml).
- 2) Carry out the operation stated in b) 2).
- 3) After allowing to cool for a while, add about 70 ml of water, and shake to mix the salts to be dissolved. Add 2 g of EDTA2Na for 0.15 g of the weighed sample, 5 g for 0.50 g and 10 g for 1.0 g, and boil for 2 min to 3 min by heating. After cooling to room temperature, add 5 ml of beryllium sulfate solution [2 j].
- 4) Adjust pH of the solution to 9.5 to 10.5 with aqueous ammonia by use of a pH meter. After boiling for 2 min to 3 min by heating, cool to room temperature. Wash the bottom surface of the watch glass with a small amount of water, remove the watch glass, filter the precipitate by use of filter paper (class 5B) to which a small amount of filter paper pulp is added, and wash 5 times to 7 times with EDTA2Na solution [2 n)]. Discard the filtrate and

washings. Transfer the precipitate together with the filter paper into the original beaker, cover it with a watch glass, add 10 ml of nitric acid and 10 ml of perchloric acid, decompose by heating, further continue to heat, and keep such state until the amount of residual solution becomes 5 ml or under ⁽¹⁾ that the vapour of perchloric acid is refluxed along the inner wall of the beaker. After allowing to cool for a while, wash the bottom surface of the watch glass with a small amount of water, remove the watch glass, add about 40 ml of water, warm, and dissolve salts by shaking ⁽²⁾.

Note ⁽²⁾ When the precipitate of tungstic acid is observed, add about 30 ml of water and 1 g of EDTA2Na, then repeat the procedure of 4) again.

- 5) After cooling to room temperature, filter the solution into a 100 ml one mark volumetric flask by use of filter paper (class 5B), wash several times with warm water, and confine washings into the filtrate. After cooling the solution to ordinary temperature, dilute to the mark with water, discard the residue.

e) Sample containing at least 0.5 % (m/m) zirconium or at least 0.1 % (m/m) niobium

- 1) Weigh a sample, and transfer to a quartz beaker (200 ml).

Informative reference : Instead of a quartz beaker, a polytetrafluoroethylene beaker with graphite base or a perfluoroalkoxy beaker can be used.

- 2) Cover with a quartz watch glass, add 15 ml of mixed acid, decompose by heating, add 5 ml of perchloric acid for 0.15 g of the weighed sample, 10 ml for 0.50 g and 15 ml for 1.0 g, continue to heat, and keep such a state for 5 min to 10 min that the inside of the beaker becomes transparent and the vapour of perchloric acid is refluxed along the inner wall of the beaker ⁽¹⁾.
- 3) After allowing to cool for a while, add approx. 40 ml of water, and shake to mix the salts to be dissolved. Add 25 ml of hydrofluoric acid (1 + 4), mix by shaking, dissolve niobic acid or the like which was deposited by heating gently, and immediately add 3.0 g of boric acid to mask excess hydrofluoric acid. After cooling to ordinary temperature, wash the bottom surface of the watch glass with a small amount of water, remove the watch glass, transfer to a 100 ml one mark quartz volumetric flask by using water, and dilute to the mark with water.

4.2 Coloration The coloration shall be in accordance with either of the following procedures.

a) In the case where sample solution is prepared in accordance with a), b), c) or d) of 4.1.

- 1) Partially take 10 ml from the solution obtained in a) 3), b) 3), c) 3) or d) 5) of 4.1, transfer into a 100 ml one mark volumetric flask ⁽³⁾, add 10 ml of sodium hydrogensulfite solution, mix by shaking, heat the solution in boiling water bath until the solution turns from red brown to almost colorless and not to vary, and keep it in boiling water bath for further 5 min.

Note (3) When a new one mark volumetric flask is used, add water to the mark, and heat for approx. 10 min in a boiling water bath. Thereafter, cool by immersing the one mark volumetric flask into running water. Repeat this operation several times, and use after variation in volume becomes little.

- 2) Add 25 ml of color reagent solution [2 o)], mix by shaking and color thoroughly by heating for 10 min to 20 min (4) in boiling water bath. Dip the one mark volumetric flask into running water, and cool to ordinary temperature. Thereafter, dilute to the mark with water.

Note (4) The heating time requires 10 min for an iron content in solution of less than 50 mg and 20 min for 50 mg to 100 mg.

- b) **In the case where sample solution is prepared in accordance with 4.1 e).**

- 1) Partially take 10 ml from the solution obtained in 4.1 e) 3), transfer to a 100 ml one mark volumetric flask (3), add 35 ml of sodium hydrogensulfite, mix by shaking, heat until the solution in boiling water bath turns from red brown to almost colorless and not to vary, and keep it in boiling water bath for further 5 min.
- 2) Add 25 ml of color reagent solution [2 o)], mix by shaking, and color it completely by heating in boiling water bath for 20 min. After cooling to ordinary temperature by dipping the one mark volumetric flask in running water, dilute to the mark with water.

4.3 Preparation of contrast solution Prepare contrast solution in accordance with either of the following procedures.

- a) **In the case where sample solution is prepared in accordance with a), b), c) or d) of 4.1.**

- 1) Partially take 10 ml from the solution obtained in a) 3), b) 3), c) 3), or d) 5) of 4.1, transfer into a 100 ml one mark volumetric flask (3), add 10 ml of sodium hydrogensulfite solution, mix by shaking, heat until the solution in boiling water bath turns to almost colorless from red brown and not to vary, and further, keep it in a boiling water bath for 5 min.
- 2) Add 25 ml of hydrazium sulfate sulfuric acid solution [2 l)], mix by shaking, and heat in boiling water bath for 10 min to 20 min (4). After cooling to ordinary temperature by dipping the one mark volumetric flask into running water, dilute the solution to the mark with water.

- b) **In the case where sample solution is prepared in accordance with 4.1 e).**

- 1) Partially take 10 ml from the solution obtained in 4.1 e) 3), transfer into a 100 ml one mark volumetric flask (3), add 35 ml of sodium hydrogensulfite solution, mix by shaking, heat until the solution in boiling water bath turns from red brown to almost colorless and not to vary, and further, keep it in boiling water bath for 5 min.

- 2) Add 25 ml of hydrazium sulfate sulfuric acid solution [2 l)], mix by shaking, and heat in boiling water bath for 20 min. After cooling to ordinary temperature by dipping the one mark volumetric flask in running water, dilute to the mark with water.

4.4 Measurement of absorbance Take each portion of the color solution obtained in 4.2 and of the contrast solution obtained in 4.3 into the absorption cell of a photometer ⁽⁵⁾, and measure the absorbance near 825 nm wavelength by employing the contrast solution as the reference solution.

Note ⁽⁵⁾ In the case where the content of phosphorus in a sample is under 0.040 % (m/m), a 2 cm absorption cell should be preferably used. In the case of 0.040% (m/m) or over phosphorus content, a 1 cm absorption cell should be preferably used.

5 Blank test Weigh the same amount of iron [2 i)] instead of the sample into a beaker ⁽⁶⁾ (200 ml), and carry out the same operation as that for the sample in parallel according to the procedure of a) 2), b) 2), c) 2), d) 2) or e) 2) of 4.1 to 4.4.

Note ⁽⁶⁾ When a sample solution is prepared in accordance with 4.1 c), use a quartz beaker.

6 Preparation of calibration graph The preparation of calibration graph shall be carried out in accordance with the following procedure.

- Prepare seven beakers ⁽⁶⁾ (200 ml) per each phosphorus content range of Annex 1 Table 2, weigh the same amount of iron [2 i)] as the weighing amount of sample in Annex 1 Table 1, and transfer it into each beaker.
- Add accurately the standard phosphorus solution [2 p)] as stated in the column of standard phosphorus solution additional amount of Annex 1 Table 2.
- Carry out the same operation as that for the sample in parallel in accordance with the procedures of a) 2), b) 2), c) 2), d) 2) or e) 2) of 4.1 to 4.4, prepare the relation curve between the obtained absorbance and the phosphorus amount added as standard phosphorus solution, transfer the relation curve in parallel so as to pass through the original point to obtain the calibration graph.

Annex 1 Table 2 Standard phosphorus solution additional amount

Phosphorus content % (m/m)	Standard phosphorus solution additional amount ml
0.005 or over to and excl. 0.040	0, 1, 2, 4, 6, 8, 10
0.040 or over to and excl. 0.15	0, 2, 4, 8, 12, 16, 20
0.15 or over up to and incl. 0.50	0, 2, 4, 8, 12, 16, 20

7 Calculation Obtain the amount of phosphorus from the absorbance obtained in 4.4 and 5 and the calibration graph prepared in 6, and calculate the content of phosphorus in a sample from the following formula :

$$P = \frac{A_1 - \left(A_2 - A_3 \times \frac{10}{100} \right)}{m \times \frac{10}{100}} \times 100$$

- where,
- P : phosphorus content in sample % (m/m)
 - A_1 : detected amount of phosphorus in aliquot sample solution (g)
 - A_2 : detected amount of phosphorus in aliquot blank test solution (g)
 - A_3 : amount of phosphorus contained in iron [2 i)] weighed in 5 (g)
 - m : weighed sample (g)

8 Precision The precision ⁽⁷⁾ shall be in accordance with any one of the following provisions :

Note ⁽⁷⁾ D in the tolerance calculation formula means $D(n, 0.95)$, and its value shall be as given in Table 4 of **JIS Z 8402**. The value of n is the number of analyses in the same laboratory for within-laboratory reproducibility and the number of laboratories related with the analyses for reproducibility (when $n = 2$, $D = 2.8$).

The (P) is the content of phosphorus [% (m/m)] in the sample for which the tolerance is obtained.

- a) The tolerance in the case where sample solution is prepared in accordance with a), b) or c) of 4.1, shall be as given in Annex 1 Table 3.

Annex 1 Table 3 Tolerance

Unit : % (m/m)

Phosphorus content	Within-laboratory reproducibility	Reproducibility
0.005 or over up to and incl. 0.10	$D_2 [0.0019 \times (P) + 0.0004]$	$D_2 [0.0059 \times (P) + 0.0009]$

Informative reference : These precisions were obtained from the results of co-operative tests by using the sample of 0.004 % (m/m) or over up to and incl. 0.088 % (m/m) phosphorus content.

- b) The tolerance in the case where sample solution is prepared in accordance with 4.1 d), shall be as given in Annex 1 Table 4.

Annex 1 Table 4 Tolerance

Unit : % (m/m)

Phosphorus content	Within-laboratory reproducibility	Reproducibility
0.005 or over up to and incl. 0.040	$D_2 [0.0043 \times (P) + 0.0004]$	$D_2 [0.0496 \times (P) + 0.0000]$