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Iron ores — Methods for determination of magnesium content

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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 M 8222:1983 is replaced with JIS M 8222:1997.

In this revision, the relevant provisions of ISO Standard have been described as Annex 2, intending the conformance with International Standard.

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Iron ores — Methods for determination of magnesium content

Introduction Annex 1 to this Standard is JIS which specifies the revised direct titration method in **JIS M 8222:1983**. Annex 2 is JIS prepared based on **ISO 10204** published in 1992 without modifying technical content and the structure of JIS.

1 Scope This Japanese Industrial Standard specifies the methods for determination of magnesium content in iron ores.

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 K 8001 *General rule for test methods of reagents*

JIS M 8202 *Iron ores—General rules for chemical analysis*

3 General matters General matters common to the determination methods shall be as specified in JIS M 8202.

4 Classification of determination methods The method for determination of magnesium content shall conform to either of the following methods.

- a) **Coexistent element separation disodium dihydrogen ethylenediamine-tetraacetate (disodium EDTA) titrimetric method** This method is applied to the sample of 0.05 % (m/m) or over up to and including 5 % (m/m) magnesium content, which is as described in Annex 1.
- b) **Flame atomic absorption spectrometric method [identical with International standard (ISO 10204)]** This method is applied to the sample of 0.01 % (m/m) or over up to and including 3.0 % (m/m) magnesium content, which is as described in Annex 2.

Annex 1 (normative) Coexistent element separation disodium EDTA titrimetric method

1 Summary Decompose a sample with hydrochloric acid and nitric acid, and filter. Extract iron in the filtrate by using 4-methyl-2-pentanone. After vaporizing silicon dioxide by treating the residue with hydrofluoric acid, fuse with potassium disulfate, and combine it with the filtrate. After neutralizing with potassium hydroxide, adjust pH by adding hydrochloric acid, generate the precipitate of hydroxide with hexamethylenetetramine, and filter. Take a portion of the filtrate. After separating coexisting elements with diethyldithiocarbamic acid sodium salt trihydrate (hereafter, referred to as "N,N-DDTC") and chloroform, adjust pH of the solution with aqueous ammonia, and titrate the total amount of calcium and magnesium with disodium dihydrogen ethylenediaminetetraacetate (hereafter, referred to as "disodium EDTA") standard solution by using Eriochrome Black T (EBT) as an indicator. Separately, titrate calcium with disodium EDTA standard solution by using calcein-thymolphthalein as an indicator, and subtract the calcium amount from the total amount.

2 Reagents The reagents shall be as follows.

- a) **Hydrochloric acid**
- b) **Hydrochloric acid [10+6(°), 1+3, 2+100]**
- c) **Nitric acid**
- d) **Hydrofluoric acid**
- e) **Sulfuric acid (1+1)**
- f) **Potassium hydroxide solution (280 g/l)**
- g) **Ammonia buffer solution** Dissolve 54 g of ammonium chloride in water, add 350 ml of aqueous ammonia and dilute it with water to 1 000 ml.
- h) **Iron solution** Dissolve 8.6 g of ammonium iron (III) sulfate with 800 ml of water and 10 ml of sulfuric acid (1+1) and dilute it with water to 1 000 ml.
- i) **Potassium disulfate**
- j) **Hexamethylenetetramine solution (250 g/l)**
- k) **Hexamethylenetetramine washing liquid (5 g/l)**
- l) **N,N-DDTC trihydrate**
- m) **4-methyl-2-pentanone**
- n) **Chloroform**

- o) **0.01 mol/l disodium EDTA standard solution** Preparation storage, and standardization of this solution shall be in accordance with 4.5 (3.3) of JIS K 8001.
- p) **0.01 mol/l potassium chloride standard solution** Dissolve 1.000 9 g calcium carbonate dried at 110 °C for about 1 h with a small amount of hydrochloric acid, transfer it into a 1 000 ml one-mark volumetric flask, and dilute it to the marked line with water. Obtain the factor of this solution as follows.

Take an aliquot of 20 ml 0.01 mol/l calcium chloride standard solution, transfer it into an Erlenmeyer flask (500 ml), add 20 ml of potassium hydroxide solution and about 0.05 g of calcein-thymolphthalein mixed indicator [2 r)], mix them by shaking and dilute it to about 200 ml by adding water. Thereafter, titrate it with 0.01 mol/l disodium EDTA standard solution until its color changes from yellow green to purple and obtain the factor of standard solution from the following formula.

$$F = \frac{V}{20}$$

where F : factor of 0.01 mol/l calcium chloride standard solution

V : used amount of 0.01 mol/l disodium EDTA standard solution (ml)

- q) **EBT methanol solution** Prepare and reserve this solution in accordance with 4.4 of JIS K 8001.
- r) **Calcein-thymolphthalein mixed indicator** Mix calcein, thymolphthalein, and potassium chloride by a ratio of 1, 1, and 100, and grind them.

Note ⁽¹⁾ Hydrochloric acid (10+6) is used for extraction of solvent, which is accurately prepared.

3 Weighing out amount of sample Weigh out 1.0 g of a sample.

4 Operation

4.1 Preparation of sample solution Prepare a sample solution as follows.

- a) Transfer the weighed-out sample into a beaker (300 ml), cover it with a watch glass, add 25 ml hydrochloric acid, at first place the beaker in the low temperature region (60 °C to 100 °C) near the edge of 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 it just before boiling. Then, add 5 ml nitric acid and evaporate the solution by further heating it. After cooling, add 5 ml to 10 ml of hydrochloric acid, heat it, add about 100 ml of warm water, mix them by shaking and dissolve soluble salts. Filter the insoluble residue by using a filter paper (Class 5B), rub the inside walls of beaker by using a rubber-tipped glass rod and transfer the adhering substance onto the filter paper. Wash the surface of filter paper with warm hydrochloric acid (2+100) heated to about 40 °C to 60 °C until the coloration of iron (III) chloride is no more of served on the filter paper. Then, wash it with warm water until acid is not observed in the washing. Transfer the

insoluble residue together with the filter paper into a platinum crucible (No. 30) to be reserved. Receive the filtrate and washings with a beaker (300 ml).

- b) Evaporate the filtrate and washings by heating, generate a film on the surface of solution and dissolve the soluble salts by adding 20 ml of hydrochloric acid (10+6). After standing to cool, transfer it into a separatory funnel (200 ml), further wash the inside walls of beaker by using hydrochloric acid (10+6) and transfer it into the separatory funnel. Add 4-methyl-2-pentanone by an amount a little exceeding the amount of sample solution and vigorously mix them by shaking for about one min. After it is separated into two layers by standing still for a while, carefully take out the lower layer of aqueous solution phase into the original beaker. Further, add 5 ml of hydrochloric acid (10+6) to the organic phase in the separatory funnel and vigorously mix them again by shaking for about 10 sec. After it is separated into two layers by standing still for a while, take out the lower layer of aqueous solution phase, put it into the original beaker and discard the upper layer of organic phase. Boil it by heating for about 5 min and vaporize most of organic solvent. Thereafter, add 5 ml of nitric acid and further evaporate it by heating until a film is generated on the surface of solution. Dissolve the soluble salts by adding 10 ml of hydrochloric acid and reserve it as a main solution.
- c) After drying the insoluble residue reserved in (a), incinerate it by ignition. Thereafter, allow it to cool. Moisten the residue on ignition with sulfuric acid (1+1), and about 5 ml of hydrofluoric acid and vaporize silicon dioxide and sulfuric acid by gently heating it. After cooling, add about 3 g of 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 allowing it to cool, put the platinum crucible into a beaker (300 ml), add 60 ml of warm water and dissolve the melt by quietly heating it. Thereafter, wash the platinum crucible, take it out and combine this solution with the main solution reserved in (b).

4.2 Hydroxide separation Add water to the sample solution obtained in 4.1 (c), to make up the volume to about 100 ml ⁽²⁾, heat it at 50 °C, neutralize it by adding potassium hydroxide solution until the initial precipitate is generated and then, adjust pH at 1.5 to 3.0 by dripping hydrochloric acid (1+3) by using a pH meter. Then, add 20 ml of hexamethylenetetramine solution, place it on a warm bath and generate the precipitate of hydroxide ⁽³⁾. Filter the precipitate with a filter paper (Class 5A) and wash it several times with hexamethylenetetramine washing liquid. Receive the filtrate and washings with a 250 ml one mark volumetric flask and cool it to ordinary room temperature. Thereafter, dilute it to the mark line. Discard the precipitate.

Notes ⁽²⁾ When most of iron has been extracted with 4-methyl-2-pentanone and further, the sample contains aluminum, titanium, etc., in small amount, 10 ml to 20 ml of iron solution [2 h)] shall be added.

⁽³⁾ The sample solution may be boiled on a hot plate for several min instead of maturing on a warm bath.

4.3 Separation of interfering elements Prepare two separatory funnels (500 ml), take an amount fixed in accordance with Annex 1 Table 1 depending on the total amount of calcium and magnesium contents from the sample solution obtained in 4.2.

transfer it into one separatory funnel, then take an amount fixed in accordance with Annex 1 Table 2 according to the content of calcium from the same sample solution, transfer it into another separatory funnel, add about 0.1 g of *N,N*-DDTC trihydrate and 50 ml of chloroform to each of them, mix them by shaking and extract the interfering elements to go into the chloroform. After standing still, discard the chloroform phase, carry out this operation again, repeat the operation until the chloroform phase turns transparent and thoroughly remove the interfering elements. Transfer each aqueous solution into a different Erlenmeyer flask (500 ml), wash the inside walls of separatory funnel with water and join them. When the aliquot amount is 50 ml, make up the volume of solution to 100 ml by adding water.

Annex 1 Table 1 Aliquot amount of sample solution

Total amount of calcium and magnesium contents % (m/m)	Aliquot amount ml
Under 2	100
2 or over	50

Annex 1 Table 2 Aliquot amount of sample solution

Calcium content % (m/m)	Aliquot amount ml
Under 2	100
2 or over	50

4.4 Titration of total amount of calcium and magnesium Regulate pH by adding 10 ml of ammonia buffer solution [2 g]) to the solution obtained from the sample solution partially taken in accordance with Annex 1 Table 1 in 4.3, add 2 or 3 drops of EBT methanol solution [2 q)], titrate it with 0.01 mol/l disodium EDTA standard solution, allow the point at which the red of solution disappears and the solution turns to distinct blue to be the end point and make the used amount of 0.01 mol/l disodium EDTA standard solution in that case the amount equivalent to the total of calcium and magnesium contents.

4.5 Titration of calcium Add 20 ml of potassium hydroxide solution and about 0.05 g of calcein-thymolphthalein mixed indicator [2 r]) to the solution obtained from the sample solution partially taken in accordance with Annex 1 Table 2 in 4.3, mix them by shaking and make up the amount of solution to about 200 ml by adding water. Thereafter, filtrate it with 0.01 mol/l disodium EDTA standard solution [2 o)], allow the point at which the color of solution changes from yellow green to purple by the last one drop of titre to be the end point ⁽⁴⁾.

Make the used amount of 0.01 mol/l disodium EDTA standard solution in that case the calcium equivalent amount.

Note ⁽⁴⁾ When the coloration with the mixed indicator is indistinct in titration, the following operation may be carried out.

Accurately add 1 ml to 3 ml of 0.01 mol/l calcium chloride standard solution [2 p)] to the solution to which calcein-thymolphthalein mixed indicator has been added, and titrate it with 0.01 mol/l disodium EDTA standard solution. In that case subtract the 0.01 mol/l disodium EDTA standard solution equivalent amount of added 0.01 mol/l calcium chloride standard solution from the previous titre of 0.01 mol/l disodium EDTA standard solution.

5 Blank test For a blank test, carry out the same operation as that for a sample in parallel with the sample according to the procedure of 4.1 to 4.5 without using the sample.

6 Calculation The calculation shall be as follows.

a) Calculation of magnesium content Calculate magnesium content in the sample from the titre obtained in 4.4, 4.5, and 5 according to the following formula.

$$Mg = \frac{[(V_1 - V_2) - (V_3 - V_4)] \times 0.000243}{m \times B} \times 100$$

where Mg : magnesium content in sample [% (m/m)]

V_1 : used amount⁽⁵⁾ of 0.01 mol/l disodium EDTA standard solution in titration of total amount of calcium and magnesium of the aliquot sample solution (ml)

V_2 : used amount⁽⁵⁾ of 0.01 mol/l disodium EDTA standard solution in titration of calcium of the aliquot sample solution (ml)

V_3 : used amount⁽⁵⁾ of 0.01 mol/l disodium EDTA standard solution in titration of total amount of calcium and magnesium in the aliquot blank test solution (ml)

V_4 : used amount⁽⁵⁾ of 0.01 mol/l disodium EDTA standard solution in titration of calcium of the aliquot blank test solution (ml)

m : weighed out amount of sample (g)

B : aliquot ratio of sample solution and blank test solution

Note ⁽⁵⁾ When the aliquot amounts of sample solution are different in operations of 4.4 and 4.5, the used amount of 0.01 mol/l disodium EDTA standard solution on the 50 ml of aliquot amount side is multiplied by two.

b) Calculation of magnesium oxide content Calculate the magnesium oxide content in the sample from the magnesium content according to the following formula.

$$MgO = 1.658 \times Mg$$

where MgO : magnesium oxide content in sample [% (m/m)]

Mg : the same as in a)