ICS 73.060.10
CCS D31

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 6730.8-2016

Iron ores - Determination of iron(II) content - Potassium dichromate titrimetric method

铁矿石 亚铁含量的测定 重铬酸钾滴定法

Issued on: October 13, 2016 **Implemented on: September 1, 2017**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;
Standardization Administration of the PRC

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Foreword

GB/T 6730 "Iron ores" is divided into dozens of parts. This Part is Part 8 of GB/T 6730. This Part is drafted in accordance with the rules given in GB/T 1.1-2009. This Part replaces GB/T 6730.8-1986 "Methods for chemical analysis of iron ores - The potassium dichromate volumetric method for the determination of iron (II) content". Compared with GB/T 6730.8-1986, in addition to editorial changes, the main technical changes of this Part are as follows: - CHANGE the name to "Iron ores - Determination of iron (II) content - Potassium dichromate titrimetric method"; - ADD contents such as "Warning", "Normative references", "Apparatus", "Test report", "Appendix B", "Appendix C"; - CHANGE the determination range from " >0.70%" to "0.700%~30.00%"; - ADD treatment method to eliminate high-valent manganese interference in samples; - Modify the weighing quantity of the test portion; - Modify the calculation formula of "calculation of analysis results"; - Carry out an interlaboratory precision joint test; USE the repeatability limit r and reproducibility limit R obtained by statistics instead of the "tolerance". This Part was proposed by China Iron and Steel Association. This Part shall be under the jurisdiction of National Technical Committee 317 on Iron Ores and Direct Reduction of Standardization Administration of China (SAC/TC 317). Main drafting organizations of this Part. Changsha Research Institute of Mining and

Metallurgy Co., Ltd., China Metallurgical Information and Standardization Institute. Main drafters of this Part. Xia Huaibin, Xu Bin, Zhang Bin, Yi Jia, Zhang Zhiyong, Sun Xiangli, Chen Shu, Yang Lin. The previous editions of the standard replaced by this Part were released as follows: - GB/T 6730.8-1986. Iron ores - Determination of iron (II) content - Potassium dichromate titrimetric method WARNING. The personnel using this Part shall have practical experience in regular laboratory work. This Part does not point out all possible safety problems. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions stipulated by relevant national regulations.

1 Scope

Part 8 of China's national series on the analysis of iron ores, determining ferrous iron by potassium dichromate titration. Ferrous iron is measured separately from total iron because the ratio between them says what the material actually is. Magnetite is an iron(II)-iron(III) oxide and hematite is purely iron(III), so the ferrous content distinguishes them and quantifies the mixture; and in a sintering or pelletising plant it is the running indicator of how completely the material has been oxidised, which governs both the fuel used and the reducibility of the product in the blast furnace. The determination has to be done on a sample dissolved without air contact, since ferrous iron oxidises in the beaker if given the chance - which is the practical difficulty of the method and the reason the procedure is prescribed in detail. This part applies to iron ores, iron concentrates, sinters and pellets over a mass fraction range of 0.700 to 30.00 per cent.

This Part of GB/T 6730 specifies the determination of iron (II) content by potassium dichromate titrimetric method. This Part applies to the determination of iron (II) content in iron ores, iron concentrates, sinters, and pellets. Determination range (mass fraction). 0.700%~30.00%.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable to this document.

GB/T 6379.1 Accuracy (trueness and precision) of measurement methods and results - Part

3 Principle

The general test portion is added with hydrochloric acid to transfer iron (II) into the solution. USE potassium dichromate titrimetric method to measure the iron content in the solution, to

obtain the iron (II) content.

4 Reagents and materials

Unless otherwise stated, in the analysis, only reagents that are confirmed as analytically pure and the grade 3 or higher distilled or deionized water that meets the requirements of GB/T 6682 or water of equivalent purity shall be used.

4.1 Sodium fluoride.

4.2 Sodium bicarbonate.

4.6 Saturated sodium bicarbonate solution.

4.7 Mixture of sulfuric phosphoric acid, 4+4+2. POUR 400 mL of phosphoric acid (rho=

1.70 g/mL), while stirring, into 400 mL of water; then slowly add 200 mL of sulfuric acid (rho=

1.84 g/mL) and mix well.

4.8 Ferric trichloride solution, 30 g/L.

4.10 Ammonium ferrous sulfate solution, about

0.03 mol/L. WEIGH

11.8 g of ammonium ferrous sulfate [(NH4)2Fe(SO4)2 - 6H2O] dissolved in a certain volume of sulfuric acid (5+95); TRANSFER to a 1000 mL volumetric flask; USE sulfuric acid (5+95) to dilute to the mark and mix well.

4.11 Sodium diphenylamine sulfonate indicator solution, 2 g/L. WEIGH

0.1 g of sodium diphenylamine sulfonate; dissolve it in 50 mL of water; ADD 2 drops~3 drops of concentrated sulfuric acid (rho=

1.84 g/mL); USE it after the solution is clear.

5 Apparatus

In the analysis, only ordinary experimental apparatus are used. The burette, one-mark volumetric flask, and one-mark pipette used shall meet the requirements of GB/T 12805, GB/T 12806, and GB/T 12808, respectively.

6 Sampling and sample preparation

6.1 Laboratory sample Laboratory samples for analysis shall be sampled and prepared in

accordance with GB/T 10322.1.The particle size shall be less than 100 μm. If the sample contains high levels of combined water or readily oxidizable substance, its particle size shall be less than 160 μm.

6.2 Predried sample Thoroughly mix laboratory samples. According to the provisions of GB/T 6730.1, dry the samples at 105 °C±2 °C; COOL to room temperature in a desiccator for later use.

7 Analytical procedures

7.1 Number of determinations The same predried sample shall be determined at least twice independently.

7.2 Test portion size According to Table 1, weigh the test portion, accurate to 0.0001 g.

7.3 Blank test DO a blank test along with the test portion. The reagents used must be taken from the same reagent bottle.

7.4 Verification test Analyze the same type of standard sample along with the test portion.

7.5 Determination

7.5.1 Determination of general test portion PLACE the test portion (see 7.2) in a 300 mL conical flask; ADD about

0.5 g of Iron (II) content (mass fraction) Test portion size

7.5.2 Determination of test portion containing metallic iron The test portion contains metallic iron. ADD 30 mL of ferric trichloride solution (see 4.8); PUT a non-magnetic metallic iron core stir bar (see 5.1) in; COVER the porcelain crucible lid. STIR on a magnetic stirrer (see 5.2) at a speed of 200 r/min for 20 min; WASH out the stir bar; ADD about

0.5 g of sodium fluoride (see 4.1), 30 mL of hydrochloric acid (see 4.3); then, follow the same steps as 7.5.1.

7.5.3 Determination of test portion containing high-valent manganese and no metallic iron

7.5.4 Determination of blank value ADD

6.00 mL of ammonium ferrous sulfate solution (see 4.10) and 25 mL of mixture of sulfuric phosphoric acid (see 4.7) to the blank solution; ADD water to a volume of about 100 mL. ADD 5 drops of sodium diphenylamine sulfonate indicator solution (see 4.11); USE potassium dichromate standard titration solution (see 4.9) to titrate to a stable purple; RECORD the volume V1 of the potassium dichromate standard titration solution consumed.

8 Analysis results and their representation