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**Iron ores - Determination of potassium, sodium,
vanadium, copper, zinc, lead, chromium, nickel, cobalt
elements - Inductively coupled plasma optical
emission spectrometry**

铁矿石 钾、钠、钒、铜、锌、铅、铬、镍、钴含量的测定

电感耦合等离子体发射光谱法

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Iron ores - Determination of potassium, sodium, vanadium, copper, zinc, lead, chromium, nickel, cobalt elements - Inductively coupled plasma optical emission spectrometry

WARNING - Persons using this Part shall have experience in formal laboratory work. This Part does not address all security issues. It is the user's responsibility to take appropriate safety and health measures and to ensure compliance with the conditions stipulated by relevant national regulations.

1 Scope

This Part of GB/T 6730 specifies the method for the determination of potassium, sodium, vanadium, copper, zinc, lead, chromium, nickel and cobalt content by inductively coupled plasma optical emission spectrometer (ICP-OES).

This Part is applicable to iron ore, iron concentrate, sinter and pellets. The range of measured element content is shown in Table 1.

2 Normative references

The following referenced documents are indispensable for the application of

this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 602, *Chemical reagent - Preparations of standard solutions for impurity*

GB/T 6379.1, *Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions*

GB/T 6379.2, *Measurement methods and results - Accuracy (trueness and precision) - Part 2: Determine the standard methods of measurement repeatability and reproducibility of the basic method*

GB/T 6682, *Water for analytical laboratory use - Specification and test methods*

GB/T 6730.1, *Iron ores - Preparation of pre-dried test samples for chemical analysis*

GB/T 8170, *Rules of rounding off for numerical values and expression and judgement of limiting values*

GB/T 10322.1, *Iron ores - Sampling and sample preparation procedures*

GB/T 12806, *Laboratory glassware - One-mark volumetric flasks*

GB/T 12808, *Laboratory glassware - One-mark pipettes*

3 Principle

Use mixed acid of hydrochloric acid, hydrofluoric acid, nitric acid and perchloric acid to decompose the specimen. Use inductively coupled plasma spectrometer to select the appropriate spectral line to measure the spectral intensity of the element to be detected. Calculate the final element content according to the calibration made from the standard solution.

4 Reagents and materials

In the analysis, unless otherwise stated, only use the confirmed guaranteed reagents and water of grade two and above in accordance with GB/T 6682.

4.1 High-purity iron: purity>99.99%.

4.2 Mixed flux: 2 portions of anhydrous sodium carbonate and 1 portion of boric acid are ground and mixed, dried and set aside for future use.

4.3 Hydrochloric acid: ρ is about 1.16g/mL.

Thoroughly mix the laboratory specimens. Use indentation to sample. Dry the specimen at $105^{\circ}\text{C}\pm 2^{\circ}\text{C}$ according to GB/T 6730.1.

7 Analysis steps

7.1 Number of determinations

In accordance with the procedures in Annex A, the same pre-dried specimen is independently determined at least twice. Take the average value.

NOTE: "Independently" means that the results of any subsequent and subsequent determinations are not affected by the results of previous determinations. In this analytical method, this condition means repeated measurements by the same operator at different times or by different operators, including the use of appropriate recalibration.

7.2 Amount of test material

Weigh about 1.0g of the pre-dried specimen (6.2), accurate to 0.0002g.

NOTE: The test material is quickly weighed to avoid re-absorbing moisture.

7.3 Blank test and verification test

7.3.1 Blank test

Conduct the blank test with the test material. Use high purity iron (see 4.1) instead of test material to test. Make sure it is consistent with the iron content in the material to be tested. All reagents shall be taken from the same reagent bottle.

7.3.2 Verification test

The same type of standard sample is analyzed along with the test material to do the verification test.

7.4 Measurement

7.4.1 Decomposition of test material

Place the test material (see 7.2) in a 100mL PTFE beaker (see 5.1). Use water to wet. Add 10mL of hydrochloric acid (see 4.3), 1mL of nitric acid (see 4.4), 4mL of hydrofluoric acid (see 4.5), 3mL of perchloric acid (see 4.6). Cover with PTFE cap. Heat on a hot plate. The temperature is controlled at around 150°C till it is dissolved completely. Open the lid and evaporate to dryness. Add 10mL of hydrochloric acid (see 4.7) and dissolve it on a discharge hot plate. After cooling, transfer to a 100mL volumetric flask (see 5.2). Use water to dilute to the scale. Mix well.

necessary, perform as many operations as possible until the obtained value is better than the value in Table 2. When the element concentration in the test solution is higher than $5000 \times \text{DL}$, RSDN is the only performance parameter to evaluate. The measured value shall be lower than the $\text{RSDN}_{\min}$ recommended in Table 2.

7.4.3 Calibration curve

The calibration solution is defined as the solution required to draw the analytical element calibration curve. Its concentration range in the solution is expressed in mass fraction (%). It depends on the performance parameters and linear sensitivity of the instrument. A minimum of 6 calibration solutions are required to cover the ranges shown in Table 1. For specimens with a narrow concentration range, the calibration solution shall include the effective area. If the element concentration in the solution is higher than $5000 \times \text{DL}$, an additional calibration curve shall be drawn to cover this range. When the calibration curve is nonlinear, it can choose a spectral line with a lower sensitivity or dilute the sample and calibration solution equally.

Table C.1 in Annex C lists the calibration curve concentrations in general. It is diluted from stock solution (see 4.9). The matrix of the calibration solution is as consistent as possible with the matrix in the solution to be tested. Generally, calculate according to the iron content in the test material which is 60%. The concentration of hydrochloric acid in the solution to be tested is 5%. If it is not a ready-made calibration solution, check its strength before use to determine if it can be used.

7.4.4 Measurement

Determine the calibration solutions in order from low concentration to high concentration. Start the measurement from the blank calibration solution. Read the reading three times for each measurement. Take the average value. Wash with water between each two tests. When determining each element, the iron content in the blank solution shall not be greater than the value in Table C.2.

NOTE 1: If the test solution exceeds the calibration curve range, the test solution and blank can be diluted, or the calibration solution can be reconstituted.

NOTE 2: Select spectral lines to see if there is any background interference. The blank solution and sample solution with iron added can be sucked and sprayed to test.

NOTE 3: After the calibration curve is completed, two-point recalibration can be used to deal with instrument drift after a period of time. See 8.3 for details.

After determining the calibration solution, determine the standard substance solution first. Then determine the sample solution. Use water to wash between each two tests. Standard substance solution and sample solution shall be

Annex B

(normative)

Spectrometer performance test

B.1 General requirements for spectrometer performance

The purpose of the performance tests given in this appendix is to provide the means. By optimizing the ICP spectrometer, different types of instruments can be used and different operating conditions can be found. This enables the use of different plasma spectrometers to ultimately produce consistent results. The whole process controls three basic parameters: detection limit (DL), background equivalent concentration (BEC) and short-term precision ($RSDN_{min}$). The best performance of the instrument to determine the element to be tested is shown in Table 2.

B.2 Definitions

B.2.1 Detection limit (DL): When the element produces a minimum concentration signal, any false background signal with a certain specified level can be considered to be exceeded. Additionally, the elemental concentration yields a signal that is three times the standard deviation of the background intensity.

B.2.2 Background equivalent concentration (BEC): The background intensity that is equal to the net signal intensity produced by the analyte concentration. It is a measure of sensitivity to a given wavelength.

B.2.3 Short-term precision (RSDN): The relative standard deviation of a series of instrument intensity readings obtained under predetermined conditions.

B.3 Reference solution

Use the three calibration solutions in Table C.1: blank solution, solution 1 (within the lowest concentration range determined in Table 1), and solution 2 for detection.

B.4 Procedure

The plasma spectrometer shall first be adjusted according to the manufacturer's recommendations and the laboratory's quantitative analysis practices. Aspirate the blank and take 10 intensity readings. Repeat this for solution 1, solution 2.

Use formula (B.1) to calculate the slope of the analytical curve:

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