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Replacing GB/T 8454-1987

Reduced ilmenite powder for welding rob - Determination of ferrous iron content - Potassium dichromate titrimetric method

焊条用还原钛铁矿粉 亚铁含量的测定 重铬酸钾滴定法

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Reduced ilmenite powder for welding rob - Determination of ferrous iron content - Potassium dichromate titrimetric method

WARNING - Personnel using this Standard shall have practical experience in formal laboratory work. This Standard does not point out all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated by the relevant national standards and regulations.

1 Scope

This Standard specifies to use potassium dichromate titrimetric method to determine ferrous iron content in reduced ilmenite powder for welding rob.

This Standard is applicable to determination of ferrous iron content in reduced ilmenite powder for welding rob. Range for determination (mass fraction): 1.00%~10.00%.

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

GB/T 6379.2, *Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement 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 & expression and*

4.10 Sulfuric acid-phosphoric acid mixed acid, 2+3+5.

4.11 Saturated boric acid solution.

4.12 Ferric chloride solution, 100g/L.

Weigh 100.0g of iron trichloride ($FeCl_3 \cdot 6H_2O$) to dissolve in 1000mL of water. Mix well. If the solution is turbid, it shall be filtered before use.

4.13 Ferrous ammonium sulfate solution, about 0.03mol/L.

Weigh 11.8g of ferrous ammonium sulfate [$(NH_3)_2Fe(SO_4)_2 \cdot 6H_2O$] to dissolve in 500mL of sulfuric acid (5+95). Use sulfuric acid (5+95) to transfer and dilute to 1000mL. Mix well.

4.14 Potassium dichromate standard titration solution, $c(1/6K_2Cr_2O_7) = 0.05000mol/L$.

Weigh 2.4518g of the reference reagent potassium dichromate, which has been dried at 150°C for 1h and cooled to room temperature in a desiccator. Dissolve in 500mL of water. Transfer to a 1000mL volumetric flask. Use water to dilute to the scale. Mix well.

4.15 Sodium diphenylamine sulfonate indicator solution, 2g/L.

Weigh 0.20g of sodium diphenylamine sulfonate to dissolve in 100mLof water. Mix well. If the solution is turbid, it shall be filtered before use.

5 Equipment and materials

Unless otherwise specified, use normal laboratory equipment. Burettes, single-marked volumetric flasks and single-marked pipettes shall meet the requirements of GB/T 12805, GB/T 12806 and GB/T 12808 respectively.

5.1 Metal iron core stirrer (non-magnetic), covered with PTFE plastic.

5.2 Adjustable speed magnetic stirrer.

5.3 Non-magnetic sample spoon.

6 Sampling and sample preparation

6.1 Laboratory specimen

Conduct sampling and sample preparation according to GB/T 10322.1. The particle size of the laboratory specimen for analysis shall be less than 74μm.

standard titration solution consumed, V_2. Add 10.00mL of ferrous ammonium sulfate solution to the solution again (see 4.13). Use potassium dichromate standard titration solution (see 4.14) to titrate till stable purple. Write down the titrated volume V_3. (V_2-V_3) is the blank of the total amount of ferrous iron and metallic iron.

7.4 Determination of metallic iron

7.4.1 Weigh 0.50g of sample, to the nearest of 0.0001g. Place in a dry 150mL Erlenmeyer flask. Add 30mL of ferric chloride solution (see 4.12). Add the magnetic core. Plug the rubber stopper. Stir on the electromagnetic stirrer for 60min.

7.4.2 Remove. Use medium speed filter paper to filter into a 250mL volumetric flask. Wash the conical flask 5 times and filter paper 5 times. Dilute to the scale. Mix well. Pipette 50.00mL of solution into a 300mL Erlenmeyer flask.

7.4.3 Add 15mL of sulfuric acid-phosphoric acid mixed acid (see 4.10) to the solution obtained in 7.4.2. Add 3 drops of sodium diphenylamine sulfonate indicator solution (see 4.15). Use potassium dichromate standard titration solution (see 4.14) to titrate till stable purple as the end point. Write down the volume of potassium dichromate standard titration solution consumed V_4.

7.4.4 Into the blank solution accompanying with the sample, accurately add 10.00mL of ferrous ammonium sulfate solution (see 4.13), 15mL of sulfuric acid-phosphoric acid mixed acid (see 4.10), and 3 drops of sodium diphenylamine sulfonate indicator solution (see 4.15). Use potassium dichromate standard titration solution (see 4.14) to titrate till stable purple. Write down the volume of potassium dichromate standard titration solution consumed V_5. Add 10.00mL of ferrous ammonium sulfate solution to the solution again (see 4.13). Use potassium dichromate standard titration solution (see 4.14) to titrate till stable purple. Write down the titrated volume V_6. (V_5-V_6) is the blank of metal iron.

8 Calculation of analysis results

8.1 Calculate the mass fraction (w_A) of the total amount of metallic iron and ferrous iron in the sample according to formula (1). The value is expressed as a percentage (%).

$$w_A = \frac{c \times [V_1 - (V_2 - V_3)] \times M}{m_0 \times 1\ 000} \times 100 \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (1)$$

Where,

c - Concentration of potassium dichromate standard titration solution, in moles

9.4 Acceptance of analysis values

The acceptance of analysis values is verified using certified reference materials (CRM) or standard samples (RM). Compare the final laboratory results with the standard value A_c. If

a) $|\mu_c - A_c| \leqslant C$, then there is no significant difference between the reported result and the standard value;

b) $|\mu_c - A_c| > C$, then there is a significant difference between the reported result and the standard value.

Where,

μ_c - CRM or RM measured value;

A_c - CRM or RM standard value;

C - Its value depends on the type of CRM/RM used.

The C value of the certification standard sample (CRM) or standard sample (RM) determined by multiple laboratories is calculated according to formula (5):

$$C = \frac{1}{\sqrt{2}} \sqrt{R^2 - \frac{n-1}{n} r^2 + 8u^2} \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (5)$$

Where,

R - Inter-laboratory reproducibility limit;

r - Repeatability limit in the laboratory;

n - Number of repeated determinations of the standard sample;

u - Uncertainty of CRM/RM sample standard value.

9.5 Calculation of final result

The final result is the arithmetic mean of the acceptable values of the specimen, or the value measured according to the operation specified in Annex A. The analysis results are revised according to GB/T 8170. The analysis result keeps two decimal places.

------ The End ------