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Iron Ores - Determination of Silicon Content - Gravimetric Methods

铁矿石 硅含量的测定 重量法

(ISO 2598-1:1992 Iron Ores - Determination of Silicon Content - Part 1: Gravimetric Methods, NEQ)

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Iron Ores - Determination of Silicon Content - Gravimetric Methods

WARNING: This Part may involve hazardous substances, operations and equipment. The personnel adopting this Part shall have practical experience in formal laboratory work. This Part does not point out all possible safety issues. It is the user's responsibility to take appropriate safety and health measures and ensure the proper scope of application before use.

1 Scope

This Part of GB/T 6730 specifies the gravimetric determination of silicon content in iron ores.

This Part is applicable to natural iron ores, iron concentrates and man-made ore agglomerates, including sintered products. The range of determination (mass fraction) is 1.00% ~ 15.00%.

Method 1: this method is not applicable to iron ores with a reducing agent content greater than 2% (mass fraction), for example, pyrite, or iron ores with fluorine content greater than 0.1% (mass fraction). This method is recommended for low-grade ores with a relatively high amphoteric element content.

Method 2: this method may be used for iron ores with fluorine content greater than 0.1% (mass fraction). This method is recommended for high-grade ores with low gangue content.

NOTE: for samples whose silicon content is less than 5% (mass fraction), the silico-molybdic acid reduction spectrophotometry is more suitable.

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 6682 Water for Analytical Laboratory Use - Specification and Test Methods (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 6730.1 Methods for Chemical Analysis of Iron Ores - Preparation of Pre-dried Test Samples for Chemical Analysis (GB/T 6730.1-1986, idt ISO 7764:1985)

GB/T 6730.3 Methods for Chemical Analysis of Iron Ores - The Gravimetric Method for

the Determination of Hygroscopic Moisture Content in Analytical Samples (GB/T 6730.3-1986, idt ISO 2596:1984)

GB/T 10322.1 Iron Ores - Sampling and Sample Preparation Procedures (GB/T 10322.1-2000, ISO 3082:1998, IDT)

GB/T 12806 Laboratory Glassware - One-mark Volumetric Flasks (GB/T 12806-2011, ISO 1042:1998, NEQ)

GB/T 12808 Laboratory Glassware - One-mark Pipettes (GB/T 12808-1991, eqv ISO 648:1977)

3 Principle

Use Method 1 or Method 2 to decompose the specimen to be tested.

Method 1: use sodium peroxide to melt and decompose, then, use hydrochloric acid and perchloric acid for treatment.

Method 2: use hydrochloric acid, nitric acid and perchloric acid (if necessary, including boric acid) to dissolve, and evaporate, until it emits perchloric acid fumes. Then, filter the silica precipitate together with all residues; use sodium carbonate to melt it, and dissolve it in hydrochloric acid and perchloric acid.

Evaporate the solution obtained through Method 1 or Method 2, until it emits perchloric acid fumes; filter the precipitated silica. Burn the impure silica and weigh it. Use hydrofluoric acid and sulfuric acid to treat the impure silica, then, burn it and weigh it again. The difference between the two weighing is the mass of the silica.

4 Reagents and Materials

Unless it is otherwise stated in the analysis, only approved analytically pure reagents and Grade-2 water that complies with the stipulations of GB/T 6682 are used in the analysis.

4.1 Sodium peroxide, solid.

4.2 Boric acid, solid.

4.3 Sodium carbonate, anhydrous.

4.4 Hydrochloric acid, ρ = 1.19 g/mL.

4.5 Hydrochloric acid, 1 + 1.

4.6 Hydrochloric acid, 1 + 9.

has evaporated. However, evaporation to dryness shall be prevented.

Let the solution cool, then, add about 25 mL of hydrochloric acid (see 4.5); stir and slowly heat it to dissolve the soluble salts. Let the precipitate settle for a few minutes. Then, use about 30 mL of water to rinse the wall of the beaker. Then, follow the operation of 7.4.2.

NOTE: the mixture shall not reach the melting point. In case that happens, it is recommended to repeat the operation at a relatively low temperature.

7.4.1.2 Acid-soluble method (Method 2)

Place the test portion (see 7.2) in a 400 mL beaker in low form; use 5 mL of water to moisten it. For ores with fluorine content greater than 0.1% (mass fraction) or unknown fluorine content, before adding 5 mL of water, firstly, add 0.8 g of boric acid (see 4.2) to the beaker containing the test portion.

Add 50 mL of hydrochloric acid (see 4.4). Use a watch glass to cover the beaker. Near the boiling point, slowly heat it, until the test portion is completely decomposed. Add 1 mL of nitric acid (see 4.11), then, add 25 mL of perchloric acid (see 4.7).

Add 1 mL of sulfuric acid (see 4.9) to prevent titanium precipitation.

Offset the watch glass on the beaker, heat it, until it emits thick white smoke of perchloric acid. Then, tightly cover the watch glass and continue heating, until there is no flowing smoke in the beaker. Maintain this stage, until most of the perchloric acid has evaporated. However, evaporation to dryness shall be prevented.

Let the solution cool, then, add about 25 mL of hydrochloric acid (see 4.5); stir and slowly heat it to dissolve the soluble salts. Let the precipitate settle for a few minutes. Add about 30 mL of water to mix it; use a small piece of filter paper or dense filter paper with a small amount of pulp to filter it.

Use a glass rod with a rubber tip to wipe off the inner wall of the beaker. Use hot hydrochloric acid (see 4.6) to wash the residue for 3 or 4 times. Finally, use hot water to wash it, until it is free of acidity.

Through the following procedures, recover the silica in the filtrate and washing solution. Add 10 mL of perchloric acid (see 4.7) and 1 mL of sulfuric acid (see 4.9) to the filtrate and washing solution; heat it, until it emits thick white smoke of perchloric acid. Then, tightly cover the watch glass and continue heating, until there is no flowing smoke in the beaker. Maintain this stage, until most of the perchloric acid has evaporated. However, evaporation to dryness shall be prevented. Repeat the procedures specified in Paragraph 5 and Paragraph 6, then, in accordance with Paragraph 2 of 7.4.2, continue the operation.

Put the residue, together with the filter paper, into a platinum crucible (see 5.2). Dry it;

ash the filter paper. Finally, burn it in a muffle furnace (see 5.4); control the temperature at 750 °C ~ 800 °C. Let the crucible cool; add 2 g ~ 3 g of sodium carbonate (see 4.3); use a nickel spoon (see 5.3) to mix it and heat it in the muffle furnace; control the temperature at 900 ~ 1,000 °C, until it is completely melted.

Let the crucible cool, then, place it in a 600 mL beaker in low form; use a watch glass to cover it. Add 200 mL of water, 50 mL of hydrochloric acid (see 4.4) and 25 mL of perchloric acid (see 4.7). Take the crucible out of the beaker, then, use hydrochloric acid (see 4.6) and water to rinse it. Use a glass rod with a rubber tip to wipe off the attachment on the inner wall of the crucible. Place the beaker on an electric heating plate to slowly heat the solution, so as to dissolve the melt.

Add 1 mL of sulfuric acid (see 4.9) to prevent titanium precipitation.

Offset the watch glass on the beaker, heat it, until it emits thick white smoke of perchloric acid. Then, tightly cover the watch glass and continue heating, until there is no flowing smoke in the beaker. Maintain this stage, until most of the perchloric acid has evaporated. However, evaporation to dryness shall be prevented.

Let the beaker cool, then, add about 25 mL of hydrochloric acid (see 4.5); stir and slowly heat it to dissolve the soluble salts. Let the precipitate settle for a few minutes. Then, use about 30 mL of water to rinse the wall of the beaker. Then, in accordance with 7.4.2, continue the operation.

7.4.2 Treatment of silica

Use a small piece of filter paper or dense filter paper with a small amount of pulp to filter the solution containing insoluble silicon in 7.4.1.1 or 7.4.1.2. Use a glass rod with a rubber tip to wipe off the wall of the beaker and use water to rinse it. Use hot hydrochloric acid (see 4.6) to wash the residue, then, use hot water to completely wash the perchloric acid. Finally, use warm water to wash it, until it is checked with silver nitrate (see 4.12) that there is no chloride ion; retain the residue on the filter paper. For ores whose silicon content exceeds 5% (mass fraction) [about 10% (mass fraction) silica] or ores with unknown silicon content, the silica in the filtrate and washing solution shall be recovered.

Place the residue, together with the filter paper, in a platinum crucible (see 5.2). Slowly heat and evaporate it to dryness, then, ash the filter paper; place it in a muffle furnace (see 5.4) with the temperature controlled at 1,050 °C ± 20 °C to burn for 30 min.

Cool it in a desiccator and weigh it as impure silicon, accurate to 0.0001 g. As described above, repeat the burning, until it reaches a constant mass (m_1). Use a few drops of water to moisten the residue in the crucible, then, add 5 drops of sulfuric acid (see 4.8). Then, in accordance with the silica content, add 5 mL ~ 15 mL of hydrofluoric acid (see 4.10). Slowly heat it in a fume hood to volatilize the silica and sulfuric acid. Evaporate it, until the white smoke of sulfur trioxide is exhausted, and repeat once. Finally, put

N_C---the number of verified laboratories;

n---the number of repeated determinations of the standard sample (in most cases, n = 1);

σ_r and σ_L are as specified in 8.2.1.

If the conditional expression (6) is satisfied, that is, the left side is less than or equal to the right side, then, $|A_C - A|$ has no statistically significant difference, otherwise, there is a statistically significant difference.

When the difference is significant, it shall be re-analyzed together with the specimen. If the difference is still significant, another standard sample of the same type of ore shall be used to repeat the operation.

When the range of the two values of the sample exceeds the limit of r calculated in accordance with Formula (3) in 8.2.1, in accordance with the flow chart in Appendix A, one or more additional analyses shall be simultaneously carried out with the standard sample of the same type of ore.

Under any circumstances, the acceptability of the analytical value of the specimen shall depend on the acceptability of the analytical value of the standard sample.

When the status of the standard sample is not fully known, the following procedure shall be adopted:

a) If there is sufficient data to estimate the inter-laboratory standard deviation, delete S_{WC}/n_{WC} and consider S_{LC} as the inter-laboratory standard deviation;

b) If there is only one laboratory for appraisal or there are no intermediate laboratory results, Formula (6) can be simplified to Formula (7).

$$|A_C - A| \leqslant 2\sqrt{2\sigma_L^2 + \frac{\sigma_r^2}{n}} \qquad \cdots\cdots(7)$$

8.2.3 Calculation of final result

The final calculation result of silica is the arithmetic mean value of the acceptable values of the specimen. Under another circumstance, determine it in accordance with the operation specified in Appendix A. Calculate it to the fourth decimal place; in accordance with the following methods, round it off to the second decimal place:

---When the leftmost digit of the number to be discarded is less than 5, then, discard it, that is, the reserved digits remain unchanged.

---When the leftmost digit of the number to be discarded is greater than 5, or equal to 5, but not all the digits after it are zero, then, advance by 1, that is, add 1 to the last reserved digit.

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