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Iron ores - Determination of total iron content - Titanium (III) chloride reduction potassium dichromate titration methods (routine methods)

铁矿石　全铁含量的测定

三氯化钛还原重铬酸钾滴定法（常规方法）

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Iron ores - Determination of total iron content - Titanium (III) chloride reduction potassium dichromate titration methods (routine methods)

WARNING: Personnel using this Part of GB/T 6730 shall have practical experience in formal laboratory work. This Part does not address all possible safety issues. It is the user's responsibility to take appropriate safety and health measures and ensure compliance with the relevant national laws and regulations.

1 Scope

This Part of GB/T 6730 specifies the method for the determination of total iron content by titanium trichloride reduction potassium dichromate titration method (conventional method).

This Part applies to the determination of total iron content in natural iron ore, iron concentrate and lump ore, including sinter and pellets. The determination range (mass fraction): 25%~72%.

2 Normative references

The following documents contain the provisions which, through reference in this Part of GB/T 6730, become the provisions of this Part. For dated references, their subsequent amendments (excluding corrigendum) or revisions do not apply to this Part. However, the parties who enter into agreement based on this Standard are encouraged to investigate whether the latest versions of these documents are applicable. For undated reference documents, the latest versions apply to this Part.

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 6379.2-2004, ISO 5725-2:1994, IDT)

GB/T 6682, *Water for analytical laboratory use - Specification and test methods* (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 6730.1, *Iron ores - Preparation of pre-dried test samples for chemical analysis* (GB/T 6730.1-1986, idt ISO 7764:1998)

GB/T 10322.1, *Iron ores - Sampling and sample preparation procedures* (GB/T 10322.1-2000, idt ISO 3082:1998)

GB/T 12805, *Laboratory glassware - Burettes* (GB/T 12805-1991, neq ISO 385:1984)

GB/T 12806, *Laboratory glassware - One-mark volumetric flasks* (GB/T 12806-1991, neq ISO 1042:1983)

GB/T 12808, *Laboratory glassware - One mark pipettes* (GB/T 12808-1991, neq ISO 648:1977)

3 Principle

According to the nature of the specimen and the content of coexisting elements, use any of the following methods to decompose the test material:

a) Decomposition by hydrochloric acid-sodium fluoride: the test material is heated and decomposed by hydrochloric acid-sodium fluoride.

b) Decomposition by sulfuric acid-phosphoric acid: the test material is heated and decomposed by sulfuric acid-phosphoric acid.

c) Melting by sodium carbonate-boric acid mixed flux, decomposition by hydrochloric acid: the test material is melted by sodium carbonate-boric acid mixed flux (full flux); the frit is heated and decomposed by hydrochloric acid.

d) Melting by sodium carbonate-sodium peroxide mixed flux, decomposition by hydrochloric acid: the test material is sintered at high temperature by a mixed flux of sodium carbonate, potassium nitrate and oxalic acid; the sintered block is decomposed by hydrochloric acid and sodium fluoride.

e) S Melting by sodium carbonate, potassium nitrate and oxalic acid mixed flux, decomposition by hydrochloric acid: the sample is sintered with sodium carbonate, potassium nitrate and oxalic acid mixed flux at high temperature, and the sintered block is decomposed with hydrochloric acid and sodium fluoride.

After the test material is decomposed, use stannous chloride to reduce most of the ferric iron in the test solution. Then use sodium tungstate as indicator. Titanium trichloride reduces all the remaining ferric iron to divalent to form "tungsten blue". Use dilute potassium dichromate solution (or carry out natural oxidation with oxygen in the air) to oxidize excess reducing agent. In sulfuric acid-phosphoric acid medium, use sodium diphenylamine sulfonate as an indicator. Use potassium dichromate standard titration solution to titrate divalent iron. Calculate the mass fraction of total iron.

When the copper content is greater than 0.5%, use any method that can completely decompose the test material to decompose the test material. Use ammonia water to precipitate iron and separate from copper. Perform determination of iron.

When the vanadium content is greater than 0.1%, use sodium carbonate-sodium

4.15 Ammonia water: 5+95.

4.16 Sodium hydroxide solution: 10 g/L.

4.17 Sodium fluoride solution: 50 g/L, stored in a plastic bottle.

4.18 Stannous chloride solution: 60 g/L.

Dissolve 6 g of stannous chloride in 20 mL of hot concentrated hydrochloric acid. Use water to dilute to 100 mL. Mix well. Add several tin particles.

4.19 Sodium tungstate solution: 250 g/L.

Weigh 25 g of sodium tungstate and dissolve it in an appropriate amount of water. Add 5 mL of phosphoric acid. Use water to dilute to 100 mL.

4.20 Titanium trichloride solution: 1+14.

Take 2 mL of titanium trichloride solution (approximately 15% mass volume concentration). Use hydrochloric acid (1+5) to dilute to 30 mL.

4.21 Copper sulfate: 5 g/L.

4.22 Potassium dichromate solution: 1 g/L.

4.23 Potassium permanganate solution: 4 g/L.

4.24 Sodium diphenylamine sulfonate indicator solution: 2 g/L.

4.25 Ammonium ferrous sulfate solution, $c(Fe^{2+}) = 0.05$ mol/L.

Weigh 19.7 g of ammonium ferrous sulfate $[(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O]$. Dissolve in sulfuric acid (5+95). Transfer to a 1000 mL volumetric flask. Use sulfuric acid (5+95) to dilute to the scale. Mix well.

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

Weigh 2.4515 g of potassium dichromate (reference reagent) that has been pre dried at 140℃~150℃ for 2 h and cooled to room temperature in a dryer. Put it in a 250 mL beaker. Add water to dissolve. Transfer to a 1000mL volumetric flask. Dilute to the scale. Mix well. Note down the temperature at which the standard titration solution is prepared.

The volumetric flasks used shall meet the requirements for Grade A volumetric flasks in GB/T 12806. Calibrate the volumetric flask beforehand if necessary.

NOTE 1: Certain potassium dichromate standard substances have determined their potassium dichromate content. The mass of potassium dichromate can be calculated and weighed according to its content.

NOTE 2: Attention shall be paid to the ambient temperature of potassium dichromate standard titration solution. The temperature during titration shall be as consistent as possible with the temperature of the prepared standard titration solution.

5 Instruments

Unless otherwise specified, the burettes, volumetric flasks and pipettes used shall comply with the provisions of GB/T 12805, GB/T 12806 and GB/T 12808.

5.1 Platinum crucible: the volume is 20 mL ~ 30 mL.

5.2 Corundum crucible: the volume is 20 mL ~ 30 mL.

5.3 Weighing spoon: it is made of non-magnetic material or demagnetized stainless steel.

5.4 High temperature furnace: the temperature is suitable to be controlled in the range of 500℃ ~ 1000℃.

6 Sampling and sample preparation

6.1 Laboratory specimens

Prepare laboratory specimens in accordance with the provisions of GB/T 10322.1. Generally, the particle size of the specimen shall be less than 100 μm. If the content of hydrated water or easily oxidized substances in the specimen is high, the particle size shall be less than 160 μm.

6.2 Pre-dried specimens

Mix the laboratory specimens thoroughly. Use fractional division method to sample. In accordance with the provisions in GB/T6730.1, dry the specimen at a temperature of 105°C ± 2°C.

7 Analysis steps

7.1 Number of determinations

For the same pre-dried specimen (6.2), conduct at least 2 independent determinations.

NOTE: "Independent" means that in the same laboratory, the same operator uses the same equipment and uses the same test method to independently test the same measured object within a short period of time.

when the fume of sulfuric acid is 5 cm ~ 6 cm away from the liquid surface.

NOTE 1: When the amount of silicon in the specimen is high, 0.5 g of sodium fluoride (4.4) can be added to aid for dissolution.

NOTE 2: For iron ore samples with high silicon and low iron content, in general, the mass fraction of total iron is less than 50%. Decomposition method by sulfuric acid-phosphoric acid shall not be used.

7.4.2.2 Cool for a little while. Add 20 mL of hydrochloric acid (4.9) along the beaker wall. Add stannous chloride solution (4.18) dropwise while hot until the test solution turns slightly yellow. Cool to room temperature. Add about 50 mL of water, 1 mL of sodium tungstate solution (4.19). Add titanium trichloride solution (4.20) dropwise until the test solution turns blue.

7.4.2.3 Add potassium dichromate solution (4.22) dropwise until the blue color disappears. Or wait until the oxygen in the air is oxidized until the blue color disappears. Add 5 drops of sodium diphenylamine sulfonate indicator solution (4.24). Use potassium dichromate standard titration solution (4.26) to titrate until the test solution turns from green to blue-green. When the last drop turns purplish red, it shall be the end point.

NOTE: Add 1~2 drops of copper sulfate solution (4.21). Shake well. Blue color immediately fades away. Titrate immediately after adding the indicator solution.

7.4.3 Melting by sodium carbonate-boric acid mixed flux, decomposition by hydrochloric acid

7.4.3.1 Put the test material (7.2) in a platinum crucible (5.1) that is filled with 2.5 g of sodium carbonate-boric acid mixed flux (4.5). Mix well. Cover with the platinum lid. Place the platinum crucible in a high-temperature furnace (5.4) at 950 °C for 10 min ~ 20 min for melting. Remove the crucible from the high temperature furnace and shake it. Cool.

7.4.3.2 Put the platinum crucible in a 300 mL beaker that is filled with 100 mL of hydrochloric acid (4.9). Cover the watch glass. Heat slowly until the test material is dissolved. During the dissolution process, add stannous chloride solution (4.18) continuously dropwise. Keep the test solution slightly yellow. Wash out the platinum crucible and lid. Transfer the test solution into a 500 mL Erlenmeyer flask. Add water to about 150 mL. Add 1 mL of sodium tungstate solution (4.19). Add titanium trichloride solution (4.20) dropwise until the test solution turns blue.

7.4.3.3 The following operations are the same as 7.4.1.3.

NOTE: If you are used to titration analysis in a beaker, you don't need to transfer it to a 500 mL Erlenmeyer flask. Carry out subsequent steps directly in the beaker.

7.4.4 Melting by sodium carbonate, sodium peroxide mixed flux

7.4.4.1 Specimens with vanadium content less than 0.1%

7.4.4.1.1 Put the test material (7.2) in the corundum crucible (5.2) that is pre-added with 0.5 g of anhydrous sodium carbonate (4.1). Add 2 g of sodium peroxide (4.2). Mix well. Use a small amount of sodium peroxide to cover the surface. Bake on a hot plate until browned. Place in the high temperature furnace (5.4). Slowly heat up from 400°C to 700°C~750°C. Melt for 5 min ~ 10 min until it is clear. Cool.

7.4.4.1.2 Put the corundum crucible in a 300 mL beaker that is filled with 100 mL of hydrochloric acid (4.9). Cover the watch glass. Heat slowly until the test material is dissolved. During the dissolution process, the stannous chloride solution (4.18) is continuously added dropwise. Keep the test solution slightly yellow. Wash out the corundum crucible. Cool to room temperature.

7.4.4.1.3 The following are the same as 7.4.1.2 and subsequent steps.

7.4.4.2 Specimens with vanadium content greater than 0.1%

7.4.4.2.1 Same as 7.4.4.1.1.

7.4.4.2.2 Put the corundum crucible in a 400 mL beaker. Add 150 mL of hot water to soak. Wash out the crucible. Use medium speed filter paper to filter. Use sodium hydroxide solution (4.16) to rinse the beaker and precipitate for 5~6 times.

7.4.4.2.3 Use 20 mL of hydrochloric acid (4.8) to dissolve the precipitate in the original beaker. Use a small amount of hot hydrochloric acid (4.10) to wash the filter paper until it is colorless. Clean the original corundum crucible. The washing liquid is combined in the original beaker. Heat slowly until the precipitate is dissolved. During the dissolution process, the stannous chloride solution (4.18) is continuously added dropwise. Keep the test solution slightly yellow. Cool to room temperature. Transfer the test solution into a 500 mL Erlenmeyer flask. Add water to about 150 mL. Add 1 mL of sodium tungstate solution (4.19). Add titanium trichloride solution (4.20) dropwise until the test solution turns blue.

7.4.4.2.4 The following operations are the same as 7.4.1.3.

NOTE: If you are used to titration analysis in a beaker, you don't need to transfer it to a 500 mL Erlenmeyer flask. Carry out the subsequent steps directly in the beaker.

7.4.5 Sintering by sodium carbonate, potassium nitrate and oxalic acid mixed flux, decomposition by hydrochloric acid

7.4.5.1 Put the test material in a porcelain crucible that is filled with 0.8 g of mixed flux of sodium carbonate, potassium nitrate and oxalic acid (4.6). Mix well. Transfer them all to a conical filter paper. Pack into a small packet. Place in a porcelain crucible that is lined with magnesium oxide (4.3). Open the furnace door in the high-temperature

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