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Replacing GB/T 6730.11-1986

Iron ores - Determination of aluminum content - EDTA titrimetric method

铁矿石 铝含量的测定 EDTA 滴定法

(ISO 6830:1986, MOD)

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Iron ores - Determination of aluminum content - EDTA titrimetric method

WARNING - Persons using this Part 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 conditions stipulated in the relevant national laws and regulations.

1 Scope

This Part of GB/T 6730 specifies the determination of aluminum content by EDTA titration.

This Part applies to the determination of aluminum content, in natural iron ore, iron concentrate, sinter and pellets; the determination range (mass fraction) is 0.25% ~ 5.0%.

2 Normative references

The provisions in following documents become the provisions of this Part of GB/T 6730 through reference in this Part. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Part; however, parties who reach an agreement based on this Part are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6682 Water for laboratory use - Specifications (GB/T 6682-1992, neq ISO 3697:1987)

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, eqv ISO 7764:1985)

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

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)

It is prepared on the day of use. The temperature of the preparation water shall be lower than 20 °C. Use fast filter paper to filter it. Then cool to 10 °C.

4.12 Chloroform.

4.13 Sodium hydroxide solution, 200 g/L. Store in polyethylene bottles.

4.14 Sodium hydroxide solution, 500 g/L. Store in polyethylene bottles.

4.15 EDTA (disodium ethylenediaminetetraacetic acid) solution, 0.04 mol/L.

Weigh 14.9 g of EDTA in 500 mL of water. Heat to dissolve. Dilute to 1000 mL after cooling. Mix well. Store in polyethylene bottles.

4.16 Sodium fluoride solution, 40 g/L.

Weigh 40 g of sodium fluoride in a polyethylene beaker. Add 1000 mL of hot water to dissolve it. Mix well (if there is precipitation, discard it after standing). Store in a polyethylene bottle.

4.17 Methyl orange indicator, 1 g/L.

4.18 Xylenol orange indicator, 1 g/L.

Dissolve 0.1 g of xylenol orange in 50 mL of water. Add 50 mL of ethanol. Mix well. Store in a brown bottle. This solution can be stored for one month, in the dark.

4.19 Acetate buffer solution

Weigh 136 g of sodium acetate ($CH_3COONa \cdot 3H_2O$). Dissolve it in about 600 mL of water. Add 7 mL of glacial acetic acid. Use water to dilute it to 1000 mL. Mix well.

4.20 Zinc standard titration solution, 0.02000 mol/L or 0.01000 mol/L.

Weigh 1.6276 g or 0.8138 g of zinc oxide (reference substance), that has been dried at 160 °C ~ 170 °C for 2 h in advance. Put it in a 300 mL glass beaker. Add 20 mL of hydrochloric acid (4.2). Heat to dissolve. Evaporate to about 5 mL. Add 200 mL of water. Add 1 drop of methyl orange indicator (4.18). Add ammonia water (4.9) dropwise to yellow. Then add hydrochloric acid (4.2) to red; add 10 drops in excess. Transfer to a 1000 mL volumetric flask. Dilute to the mark. Mix well.

5 Instruments

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

Common laboratory instruments and:

7.3.2 Verification test

Analyze the same type of standard sample, along with the sample, to do verification test.

7.4 Determination

7.4.1 Decomposition of samples

Put the sample (7.2) in a 300 mL glass beaker. Add 20 mL of hydrochloric acid (4.2). Cover the watch glass, (if the fluorine content of the sample exceeds 2 mg, use a 200 mL polytetrafluoroethylene beaker; do not cover the watch glass when dissolving the sample). Heat and decompose at low temperature for about 30 minutes. Take it out and cool slightly. Add 10 mL of nitric acid (4.5) and 3 mL of perchloric acid (4.6). Continue heating, until white fumes of perchloric acid appear. Evaporate to dryness. Add 20 mL of hydrochloric acid (4.3). Heat to dissolve. Add 20 mL of water. Stir it. Use slow filter paper to filter it. Transfer all residues in the beaker to filter paper. Use hydrochloric acid (4.4), to wash the beaker and filter paper 4 times. Then use hot water to wash 2 times. Store the filtrate and washing solution.

Ash and burn the residue, in a platinum crucible at 700 °C. Remove and cool it. Add 3 ~ 5 drops of sulfuric acid (4.8) and 5 mL of hydrofluoric acid (4.7). Heat at low temperature, until white smoke of sulfuric acid appears. Evaporate to dryness. Then remove it. Add 3 g of potassium pyrosulfate (4.1), to melt at 650 °C ~ 700 °C for 10 min. Remove and cool it. Put it into the filtrate. Heat it for leaching. Rinse out the crucible. Heat the solution, until all the melt is dissolved.

7.4.2 Separation of interfering elements

7.4.2.1 Ammonia water separation

Use water to adjust the solution to about 100 mL. Heat to boil. Add ammonia water (4.9) dropwise to pH 6.5 ~ 7.0, to completely precipitate the hydroxide. Boil for 1 min. Use fast filter paper to filter immediately. Use hot ammonium chloride solution (4.10), to wash the beaker and filter paper 7 ~ 8 times. Discard the solution. Place the unfolded filter paper and hydroxide precipitate on the inner wall of the original beaker. Use the hot water in the washing bottle, to wash off the hydroxide precipitate. Then use 25 mL hot hydrochloric acid (4.3), to wash the filter paper. Then use hot water filter paper to rinse it, until colorless. Discard the filter paper. Boil the solution. Cool it to below 20 °C. Control it at about 50 mL.

Note: Make sure that the temperature of the solution and all reagents used is below 20 °C, before and during the extraction.

7.4.2.2 Extraction and separation of copper and iron reagents

Transfer the solution to a 250 mL separatory funnel. Use water to dilute it to about 75 mL. Add 20 mL of copper-iron reagent solution (4.11). Shake gently. Add 20 mL of chloroform (4.12). Shake vigorously for 1 min. Let it stand for layering. Discard the organic phase. In order to remove the copper and iron reagents on the surface of the water phase, add another 5 mL of chloroform (4.12) to the separatory funnel. Discard the organic phase. If the iron content of the sample is less than or equal to 150 mg, repeat the above operation once. If the iron content of the sample is greater than 150 mg, repeat the above operation twice.

Add 20 mL of chloroform (4.12) to the separatory funnel. Shake vigorously for 1 min. Let it stand for layering. Discard the organic phase.

Put the aqueous phase into a 300 mL glass beaker or 300 mL conical flask. Use a small amount of water, to wash the separatory funnel. Incorporate it into the aqueous phase. Heat the solution, to evaporate about 20 mL. Add 10 mL of nitric acid and 3 mL of perchloric acid. Continue heating, until white fumes of perchloric acid appears. Evaporate to dryness. Remove and cool it. Add 10 mL of hydrochloric acid (4.3) and heat to dissolve. Evaporate to 3 mL ~ 5 mL. Add 40 mL of water. Boil it.

7.4.2.3 Strong base separation

If the sample contains rare earth elements (greater than 0.60 mg), after adding 40 mL of water, add 20 mL of LEDTA solution (4.15) and 1 drop of methyl orange indicator (4.17). Add dropwise sodium hydroxide solution (4.13), until the solution turns yellow. Then add hydrochloric acid (4.3) dropwise and the solution turns red. Add 5 drops in excess. Boil it. Add 12 mL of sodium hydroxide solution (4.14) while stirring. Leave it for 20 min. Cool it under running water. Transfer to a 100 mL volumetric flask. Dilute to the mark. Shake well. Dry filter it. Use a pipette to divide 50.00 mL of the filtrate into a 300 mL glass beaker or a 300 mL conical flask. Add hydrochloric acid (4.3) solution to turn red. Use water to dilute it to a volume of about 100 mL.

Note: This analysis step is only for iron ore containing rare earth elements (greater than 0.60 mg).

7.4.3 Titration

Add the solution (7.4.2.2) into excess EDTA solution (4.15) (usually 10 mL ~ 20 mL is enough) and 1 drop of methyl orange indicator (4.17). Add sodium hydroxide solution dropwise (4.13), until the solution turns yellow. Then add dropwise hydrochloric acid (4.3), until the solution turns red. Use water to dilute it, to a volume of about 100 mL.

Note: If the sample is separated by strong alkali, the above steps are omitted. Directly use the solution (7.4.2.3), to transfer to the following operation.

Heat to boiling. Add 15 mL of acetate buffer solution (4.19) while hot. Boil slightly for 3 min. Cool to room temperature under running water. Add 5 ~ 7 drops of xylenol orange indicator (4.18). Use zinc standard titration solution (4.20), to titrate it to red (do

- In the laboratory, it is calculated according to the formulas (2) and (4), which is the arithmetic mean of the two repeated measurement results;

- Between laboratories, it is calculated according to formulas (3) and (5), which is the arithmetic mean of the final results (8.2.5) of the two laboratories.

R_d - The allowable difference (repeatability) of repeated determinations in the laboratory;

P - The allowable difference between laboratories;

σ_d - The standard deviation of repeated determinations within the laboratory;

σ_L - The standard deviation between laboratories.

8.2.2 Determination of analysis results

Follow the steps in Appendix A, to calculate the independent repeated measurement results, according to the formula (1). Compare them with the repeated measurement tolerance (R_d), to determine the analysis results.

8.2.3 Interlaboratory precision

Interlaboratory precision is used to evaluate the consistency -- between the final results reported by two laboratories. After both laboratories report the results following the same procedure as specified in 8.2.2, make calculation as follows:

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2}$$

Where:

μ_1 - The final result reported by laboratory 1;

μ_2 - The final result reported by laboratory 2;

μ_{12} - Average of final results.

If $|\mu_1 - \mu_2| \leq P$ (see 8.2.1), the final result is consistent.

8.2.4 Acceptance of analytical values

Acceptance of analytical values is verified, using certified reference samples. The steps are the same as described above. After confirming the precision, the laboratory's final result is compared with the standard value Ac, if:

a) $|\mu_c - Ac| \leq C$, there is no significant difference between the measured value and the standard value;

b) |μc - Ac| > C, there is a significant difference between the measured value and the standard value.

Where:

μ_c - The measured value of the standard sample;

Ac - The standard value of standard sample;

C - The value depends on the type of standard samples used.

For standard samples, which are determined by interlaboratory:

$$C = 2\sqrt{\sigma_L^2 + \frac{\sigma_d^2}{n} + V(Ac)}$$

Where V(Ac) is the variance of the standard value Ac.

8.2.5 Calculation of final results

The final result is the arithmetic mean of the acceptable values of the specimens, OR, in the other case, determined by the operation specified in Appendix A. Calculate to four decimal places; round to two decimal places as follows:

a) When the third decimal place is less than 5, round off this number; the second decimal place remains unchanged;

b) When the third decimal digit is 5 and the fourth decimal digit is not 0, OR when the third decimal digit is greater than 5, the second decimal digit is rounded up by 1;

c) When the third decimal digit is 5 and the fourth decimal digit is 0, round off. If the second decimal digit is 0, 2, 4, 6, or 8, it will remain unchanged. If it is 1, 3, 5, 7, 9, round up by 1.

8.3 Oxidation coefficient

$$w_{Al_2O_3}(\%) = 1.889\,5 \times w_{Al}(\%)$$

9 Test report

The test report shall include the following information:

a) The name and address of the testing laboratory;

b) The date of release of the test report;

c) The number of this Part;

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