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**Methods for chemical analysis and physical properties test of
cryolite - Part 4: Determination of aluminum content -
EDTA titration method**

冰晶石化学分析方法和物理性能测定方法 第4部分：铝含量的测定

EDTA 滴定法

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Methods for chemical analysis and physical properties test of cryolite - Part 4: Determination of aluminum content - EDTA titration method

1 Scope

This Part specifies the method for determination of aluminum content in cryolite.

This section applies to the determination of aluminum content in cryolite. Measurement range: 9.00% ~ 20.00%.

2 Method summary

The sample is melted with acidic potassium sulfate or potassium pyrosulfate, to remove fluorine; the frit is dissolved with hydrochloric acid. Take a portion of the test solution. Add excess Na_2EDTA , to form a complex between the aluminum element and EDTA. Under weakly acidic conditions, use xylanol orange as an indicator. Use zinc nitrate standard titration solution, to titrate the excess Na_2EDTA . Add sodium fluoride. Heat to displace EDTA. Then use zinc nitrate standard solution, to titrate the released EDTA. Calculate the aluminum content by using the millimeters of zinc nitrate standard titration solution, which is consumed in the titration.

3 Reagents

Unless otherwise stated, only reagents and deionized water determined to be of analytical grade are used in the analysis.

3.1 Acid potassium sulfate or potassium pyrosulfate.

3.2 Sodium fluoride.

3.3 Hydrochloric acid (1 + 1).

3.4 Hydrochloric acid (1 + 3).

3.5 Sodium hydroxide solution (100 g/L).

3.6 Buffer solution (pH value 5.6): Weigh 333 g sodium acetate trihydrate. Dissolve in water. Add 15 mL of glacial acetic acid. Use water to dilute it to 1000 mL. Mix well.

5 Specimens

Grind and mix the sample, to pass through a 75 μm standard sieve. Bake it in an oven, at $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 2 h. Cool it to room temperature in a desiccator.

6 Analysis steps

6.1 Sample

Weigh 0.25 g of specimen (5), accurate to 0.0001 g.

6.2 Parallel test

Conduct two tests in parallel. Take the average value.

6.3 Determination

6.3.1 Place the sample (6.1) in the platinum crucible (4.1). Add 6 g of acidic potassium sulfate or potassium pyrosulfate (3.1). Put the platinum crucible into the high-temperature furnace (4.2) when the temperature is lower than $300\text{ }^{\circ}\text{C}$. Start timekeeping, when the furnace temperature reaches $725\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$. Melt for about 30 minutes, until the fluorine is completely driven out. Take it out. Cool it to room temperature.

6.3.2 Add 100 mL of water and 5 mL of hydrochloric acid (3.3) in a 250 mL beaker. Then put the frit (6.3.1) together with the platinum crucible, into the beaker. Heat it. Use a glass rod to stir, to make the frit fall off. Wash out the platinum crucible. Continue heating, until the frit is completely dissolved. Remove and cool to room temperature. Transfer the solution into a 250 mL volumetric flask. Use water to dilute it to the mark. Mix well.

6.3.3 Pipette 100.00 mL of solution (6.3.2). Add 1 drop of phenolphthalein ethanol solution (3.9). Dropwise add sodium hydroxide solution (3.5), until the solution turns red. Dropwise add hydrochloric acid (3.4), until the solution is colorless. Add an excess of 1.5 mL. Add 15 mL of Na_2EDTA solution (3.7). Heat and boil for 2 minutes. Remove it. Add sodium hydroxide solution (3.5) dropwise, until the solution turns red. Add hydrochloric acid (3.4) dropwise, until the solution is colorless. Add 20 mL of buffer solution (3.6). Use running water to cool to room temperature.

6.3.4 Add 3 drops of xylenol orange solution (3.10). Use zinc nitrate standard titration solution (3.8) for titration, to make the solution change from yellow to rose red.

6.3.5 Add 0.5 g ~ 1 g of sodium fluoride (3.2). Heat and boil for 2 minutes. Then remove it. Use running water, to cool to room temperature. Use zinc nitrate standard titration solution (3.8) for titration, until the solution changes from yellow to rose red. Record the volume of zinc nitrate standard titration solution, which is consumed in the second

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