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**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YS/T 509.9-2008

**Methods for chemical analysis of spodumene and lepidolite
concentrates. Determination of fluorine content. Ion selective
electrode method**

(English Translation)

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Foreword

YS/T 509 Methods for chemical analysis of spodumene and lepidolite concentrates consists of the following 11 parts:

- Part 1: Determination of lithium oxide sodium oxide and potassium oxide contents - Flame atomic absorption spectrometric method;
- Part 2: Determination of rubidium oxide and caesium oxide contents - Flame atomic absorption spectrometric method;
- Part 3: Determination of silicon dioxide content - Gravimetric-molybdenum blue photometric method;
- Part 4: Determination of aluminum oxide content - EDTA compleximetric method;
- Part 5: Determination of ferric oxide content - O-phenanthroline photometric method and EDTA compleximetric method;
- Part 6: Determination of phosphorus pentoxide content - The molybdenum blue photometric method;
- Part 7: Determination of beryllium oxide content - Chromazurol S and cetyltrimethyl ammonium bromide spectrophotometric met;
- Part 8: Determination of calcium oxide and magnesium oxide content - Flame

atomic absorption spectrometric method;

--- Part 9: Determination of fluorine content - Ion selective electrode method;

--- Part 10: Determination of manganous oxide content-Persulfate oxidation photometric method;

--- Part 11: Determination of loss on ignition - Gravimetric method.

This Part is Part 9.

This Part replaced YS/T 509.10-2006 Methods for Chemical Analysis of Spodumene and Lepidolite Concentrates - The Ion Selective Electrode Method for the Determination of Fluorine Content in Lepidolite Concentrates (original GB/T 3885.10-1983).

Compared with YS/T 509.10-2006, the major changes of this Part are as follows:

--- Re-edit the text format;

Methods for Chemical Analysis of Spodumene
and Lepidolite Concentrates - Determination of
Fluorine Content - Ion Selective Electrode Method

1 Scope

This Part specifies the determination method of fluorine content in spodumene and lepidolite concentrates.

This Part is applicable to the determination of fluorine content in spodumene and lepidolite concentrates. Measurement range: 1.00%~10.00%.

2 Summary of Method

The test material is melted and decomposed with sodium hydroxide and sodium peroxide. In the sodium citrate-potassium nitrate complex buffer solution with pH 7, the standard addition method is used to determine fluorine with a fluoride ion selective electrode.

Aluminum seriously interferes with the determination of fluorine. Add the sodium citrate-potassium nitrate mixed solution to the alkaline solution and adjust to pH 7, which can eliminate the interference of 10mg of aluminum oxide.

3 Reagents

Unless otherwise specified, only reagents and distilled water confirmed to be analytically pure or water of equivalent purity are used in the analysis.

6.1 Test material

Weigh 0.20g of specimen, accurate to 0.0001g.

6.2 The number of determinations

Make parallel determinations of two portions of test materials, and take the average value.

6.3 Blank test

Do a blank test with the test materials.

6.4.1 Place the test material (6.1) in a 30mL nickel crucible; add 3g of sodium hydroxide

(3.2) and 0.5g of sodium peroxide (3.1); drive off the water on the electric furnace. And place it in a high temperature furnace at 600°C~650°C for 5min~10min to melt; take it out; and cool.

6.4.2 Clean the outer wall of the crucible with filter paper; place it in a 250mL beaker;

cover the watch glass; soak the melt with hot water; wash the crucible; take it out; and cool the solution to room temperature.

6.4.3 Transfer the solution (6.4.2) into a 250mL volumetric flask; dilute to the mark with

water; mix well; and let stand.

6.4.4 Pipette 10.00mL of supernatant (6.4.3); put it in a 50mL beaker; add 10mL of

sodium citrate-potassium nitrate mixed solution (3.6); adjust the pH to 7 with hydrochloric acid (3.4 and 3.5) and sodium hydroxide solution (3.3) on the acidimeter. Then transfer the solution into a 50mL volumetric flask, dilute to the mark with water

and mix well.

6.4.5 Pour all the test solution (6.4.4) into a 100mL dry beaker; place it on the electromagnetic stirrer; insert the fluorine electrode and calomel electrode; switch on the ion meter; measure its dynamic equilibrium potential' and record millivolt value (E1). Then add 0.20mL~0.50mL of fluorine standard solution (3.7.1) with a micropipette; measure its dynamic equilibrium potential; and record the millivolt value (E2). The added amount of fluorine standard solution (3.7.1) shall be such that the value of E1-E2 is between 15mV~40mV.

NOTE 1: During the measurement process, the temperature shall be kept consistent.

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