



Determination of fluoride and chloride in alumina

Introduction:

Alumina has many good properties, and its applications are extremely wide, such as biomedical engineering materials, fine ceramics, alumina fiber high-strength and heat-resistant products, special refractory materials, catalysts and carriers, transparent alumina ceramics, flame retardants, etc. Inorganic cations are often used in the determination of impurity elements in alumina, and most of the methods used are spectra. In this paper, a simple sample pretreatment and ion chromatography are used to determine fluoride and chloride in aluminum cyanide. It has been applied to the analysis of practical samples with good results.

Keywords: Alumina, Ion chromatography, Fluoride, Chloride.

Instruments and equipment

- **Ion chromatograph:** CIC-D160
 - **Ultra pure water machine:** ECO-S15
- Qingdao Shenghan Chromatograph Technology Co., Ltd



Requirements

Reagents

All reagents used are superior grade pure or better, Purchase certified standard solutions F⁻, Cl⁻ standard solutions (1000 mg / L).

Deionized Water

When preparing standard samples manually or diluting real samples, please use ASTM filtration and deionization requirements that meet the specifications listed in the table 1.

Table 1: Deionized water specification.

Specification	
Ions Resistivity	≥18.25MΩ·cm
Organics-TOC	<10ppb
Iron/Transition Metals	<1ppb
Pyrogens	<0.03Eu/mL
Particulates (>0.2μm)	<1unit/mL
Colloids-Silica	<10ppb
Bacteria	<1cfu/mL

Sample preparation

Weigh an appropriate amount of sample, add an appropriate amount of ultrapure water, and conduct ultrasonic treatment. The sample passes through the activated Na column, and then passes through 0.22 μ M filter membrane, constant volume, sample preparation completed.

(Activation method of Na pretreatment column: pass 10 ml of deionized water, place it for 10 minutes)

Table 2: Sample preparation

Sample name	Weight (g)	Absorbent	Constant volume(mL)	Test items
1#	0.5678	Deionized water	50	F ⁻ , Cl ⁻
2#	0.4735	Deionized water	50	F ⁻ , Cl ⁻

Chromatographic conditions

Eluent: 12 mM KOH (EG)

Flow rate: 1.0 mL/min

Injection volume: 25 μL

Guard column: SH-G-1

Analytical Column: SH-AC-11

Column oven temperature: 35°C

Conductivity cell temperature: 35°C

Suppressor current: 50 mA

Standard chromatogram

Standard chromatogram, As shown in below:

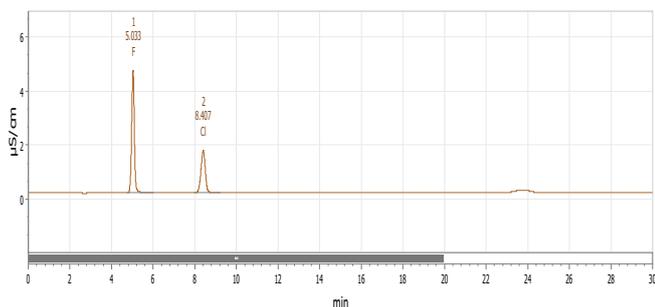


Figure 1. Chromatogram of standard sample.

Table 3: Data of standard solution

Compound name	Retention time[min]	Concentration[mg/L]	Area[(μS/cm)*min]	Height[μS/cm]
F ⁻	5.033333	1.000000	0.690102	4.554952
Cl ⁻	8.406667	1.000000	0.381253	1.56263

Comparison testing (blank)

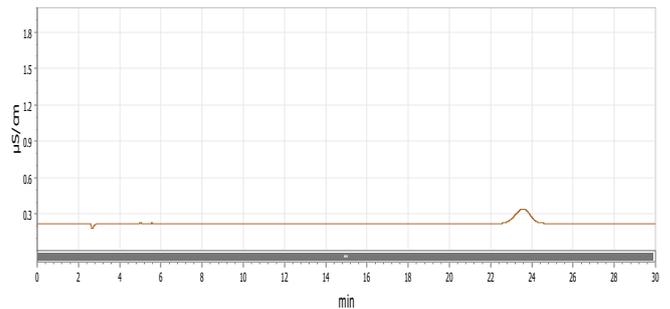


Figure 2. Blank chromatogram

Sample chromatogram

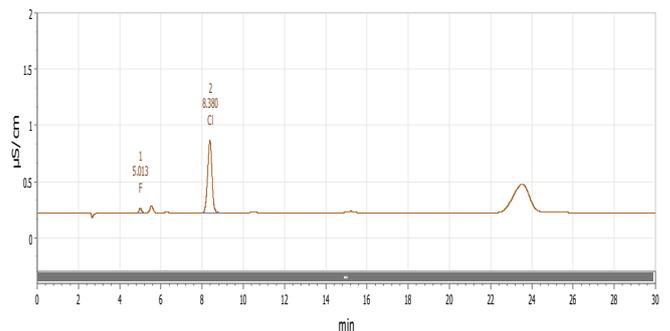


Figure 3. Chromatogram of sample 1#

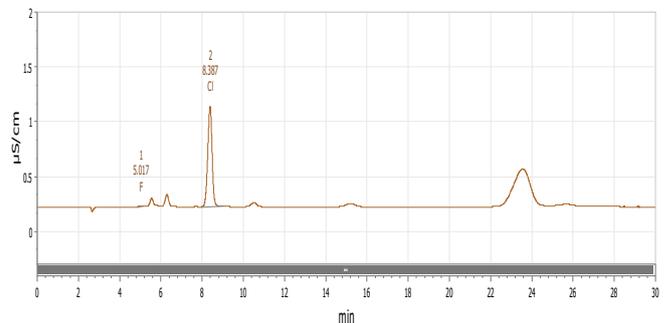


Figure 4. Chromatogram of sample 2#

Results and calculations

Table 4: Sample test results (mg/kg)

Sample	F ⁻	Cl ⁻
1#	0.6579	33.03
2#	0.1503	55.97

Remarks: ① the blank value has been deducted from the measured value; ② Different methods and different laboratories may have different test results.

Precautions

It is easy to be polluted in the process of the experiment, and the experimental personnel are required to operate in strict accordance with the operating procedures.

Feasibility analysis and conclusion

Through the above experiments, it is proved that the detection method has good separation and is suitable for the determination of the content of the components to be measured in the sample.