Detection of fluoride and chloride in germanium ores

Introduction:

Germanium has various special properties and has extensive and important applications in fields such as semiconductors, aerospace measurement and control, nuclear physics detection, fiber optic communication, infrared optics, solar cells, chemical catalysts, biomedicine, etc. It is an important strategic resource. In the electronic industry, in alloy pretreatment, and in the optical industry, it can also serve as a catalyst.

Germanium products will further increase technological innovation and transformation efforts, optimize process flow, and improve resource recovery and utilization rates. Due to its extensive demand and industrial upgrading and transformation, there is currently a demand for quality testing of germanium ore. The content of fluorine and chlorine in germanium ore is an important indicator, as fluorine and chlorine not only pollute the environment, but also corrode equipment and pipelines during the refining process, affecting product quality. Therefore, the research on the detection and methods of fluoride and chloride content in germanium ore has attracted widespread attention.

Detection items (Table 1):

Anion	F^-	Cl^-

Keywords: On-line ion chromatography, Fluoride, Chloride

Instruments and equipment

- **Ion chromatograph**: SH-CIC-3200
- **Ultra pure water machine**:ECO-S15

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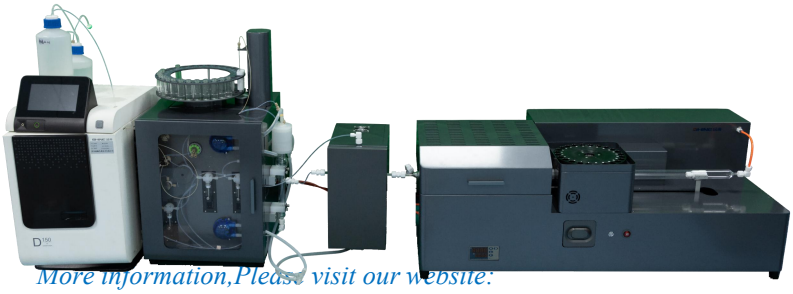

Requirements

Reagents

Unless otherwise specified, all reagents used are superior grade. F^- Cl^- anions standard solution (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 2.

Table 2: 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

Chromatography conditions (Anions):

Table 3: Anions analysis conditions

Instrument	SH-CIC-3200
Eluent	15 mM KOH
Flow rate	1.0 mL/min
Injection volume	25 μL
Analytical Column	SH-AC-23
Column oven temperature	35℃
Conductivity cell temperature	35℃
Suppressor current	45 mA

Sample preparation

First, grind the sample to a particle size not greater than 0.080 mm, and dry the sieved sample in a 105 ℃± 2 ℃ oven for 1 hour. After removal, cool it to room temperature in a dryer for later use.

Table 4:Sample preparation

No.	Weight（g）	Volume (mL)	Diluting solvent
1#	0.0998	100	NaOH solution
2#	0.0997	100	NaOH solution

Standard chromatogram

Standard chromatogram, As shown in below:

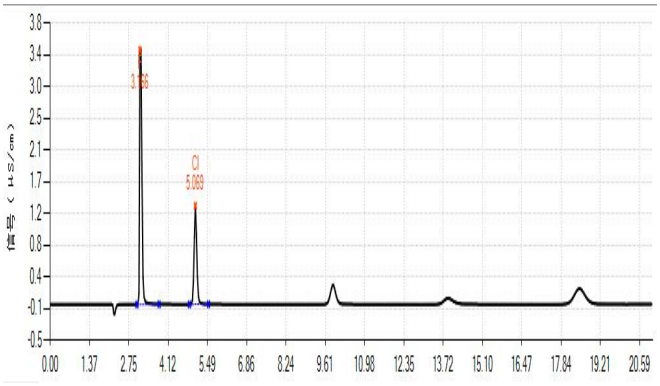

Figure 1. Chromatogram of standard sample.

Blank chromatogram

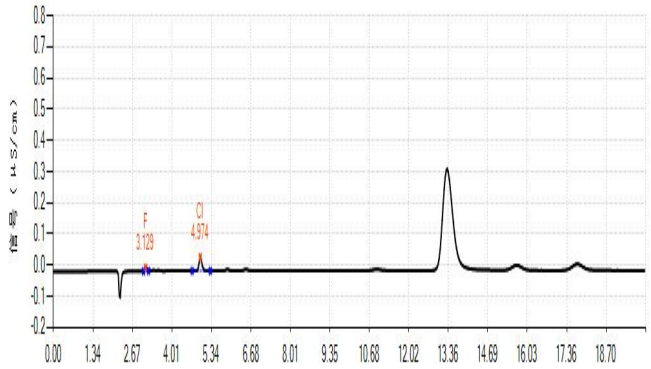

Figure 2. Chromatogram of blank

Sample chromatogram

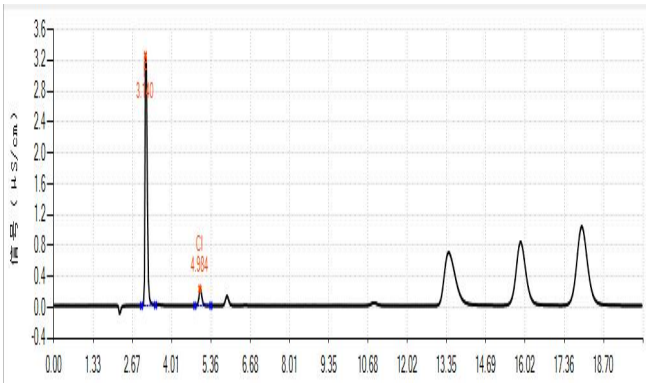

Figure 3. Chromatogram of sample 1#

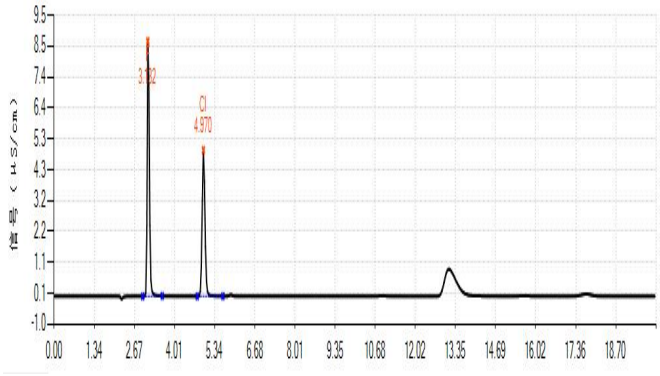

Figure 4. Chromatogram of sample 2#

Results and calculations

Table 4: Sample test result

Sample	F^-（mg/L）	Cl^-（mg/L）
1#	474.7	64.23
2#	1780	1972

Remarks: ① The measured value has been deducted from the blank value; ② There may be differences in testing results between different methods and laboratories.

Feasibility analysis and conclusion

The above experiments prove that the detection method has good resolution and is suitable for the determination of the content of the components to be measured in the sample.