Detection of anions in fire explosive materials

Introduction:

In recent years, there have been cases of criminals using inorganic explosives such as ammonium nitrate, pyrotechnic agents, and black powder for criminal activities. By examining explosives and their explosive residues, the type of explosives and the nature of the case can be determined, providing direction and clues for investigation. At present, the main detection methods in China are chemical methods, which are cumbersome, low sensitivity, and have many interferences. Traditional methods have significant limitations. Ion chromatography, as a liquid phase branch, currently has multiple applications in environmental, food and other methods, and only requires one injection and can detect multiple anions and cations. The pre-treatment of the method is simple.

Detection items (Table 1):

Anion	Cl^-	ClO_3^-	NO_3^-	SO_4^{2-}	ClO_4^-

Keywords: Anions， Ion chromatography, Explosive.

Instruments and equipment

- **Ion chromatograph**: CIC-D160$^+$
- **Ultra pure water machine**:ECO-S15

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Requirements

Reagents

Unless otherwise specified, all reagents used are superior grade. Cl^-, ClO_3^- ,NO_3^- ,SO_4^{2-} and ClO_4^-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:

Table 3: Analysis conditions

Instrument	CIC-D160$^+$
Eluent	7.0 mmol/L Na2CO3+6.0 mmol/L NaHCO3
Flow rate	0.5 mL/min
Injection volume	500 μL
Analytical Column	SH-AC-12C
Column oven temperature	35℃
Conductivity cell temperature	35℃
Suppressor current	35 mA

Sample preparation

Extract the dust at the center of the explosion and extract a series of explosion dust in a certain distance, direction, and area from the explosion point. Simultaneously extract a series of blank control samples.
Weigh 1.5g of the sample into a beaker, dissolve it in a small amount of ultrapure water, transfer it to a 100mL volumetric flask, add ultrapure water to volume, shake well, take 1mL from it into a 500mL volumetric flask, add ultrapure water to volume, shake well, After pass 0.22 μm microporous membrane, analyze it on the machine.

Standard chromatogram

Standard chromatogram, As shown in below:

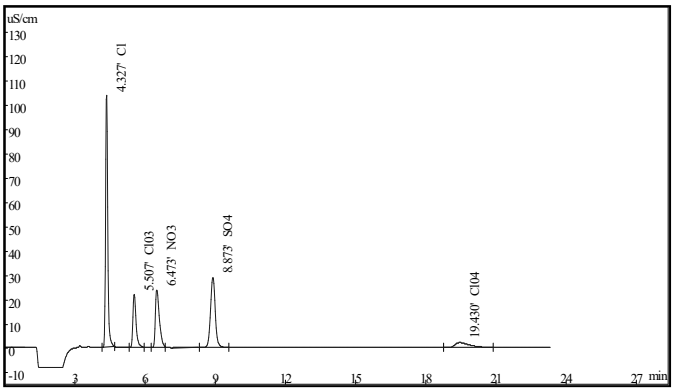

Figure 1. Chromatogram of standard sample.

Table 4: Resolution of standard sample

No.	Anions	Concentration mg/L	Area/ μs/cm*s	Resolution
1	Cl^-	1.0	582.34	6.22
2	ClO_3^-	1.0	187.02	3.73
3	NO_3^-	1.0	257.35	7.50
4	SO_4^{2-}	1.0	377.48	13.97
5	ClO_4^-	1.0	89.07	0.00

Table 5: Linearity of standard sample

Ions	Concentration/ mg/L	Linear equation	Linearity coefficient
Cl^-	0~1.0	$Y=5.544*10^8X+1692000$	0.9991
ClO_3^-	0~1.0	$Y=1.866*10^8X-1073000$	0.9998
NO_3^-	0~1.0	$Y=2.557*10^8X-728200$	0.9996
SO_4^{2-}	0~1.0	$Y=3.658*10^8X+1.128000$	0.9994
ClO_4^-	0~1.0	$Y=8.986*10^7X-441200$	0.9997

Minimum detectable concentration

Stable for 30 minutes after startup, baseline noise is 1.379ns/cm.
Calculate the theoretical minimum detection concentration with a signal-to-noise ratio of 3 times, and the theoretical minimum quantitative concentration with a signal-to-noise ratio of 10 times.

Table 6: Minimum detectable concentration

Ions	Theoretical	Theoretical

	minimum detection concentration	minimum quantitative concentration
Cl^-	0.03777	0.1259
ClO_3^-	0.17997	0.5999
NO_3^-	0.16477	0.5492
SO_4^{2-}	0.13622	0.4541
ClO_4^-	0.19108	0.6369

Sample chromatogram

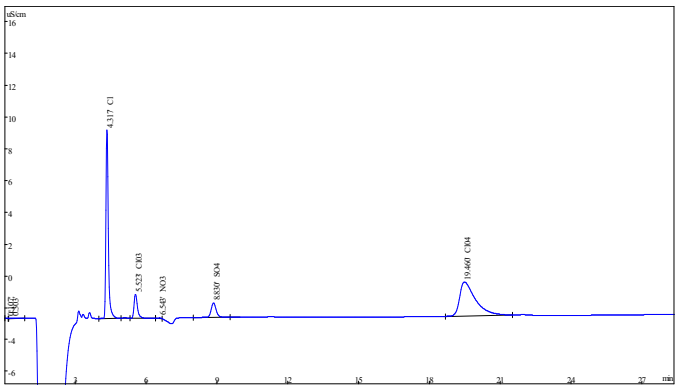

Figure 2. Chromatogram of sample

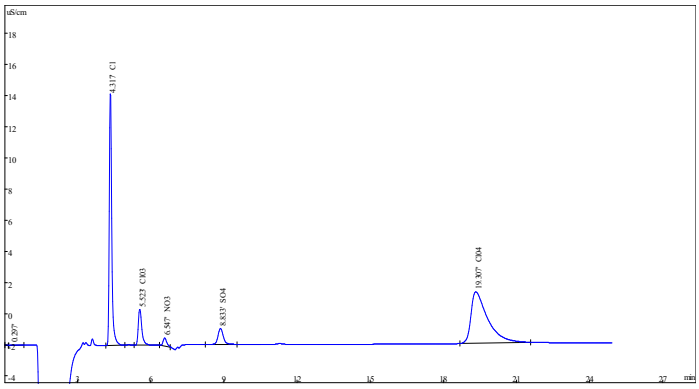

Figure 3. Sample added scalar quantity0.05mg/kg

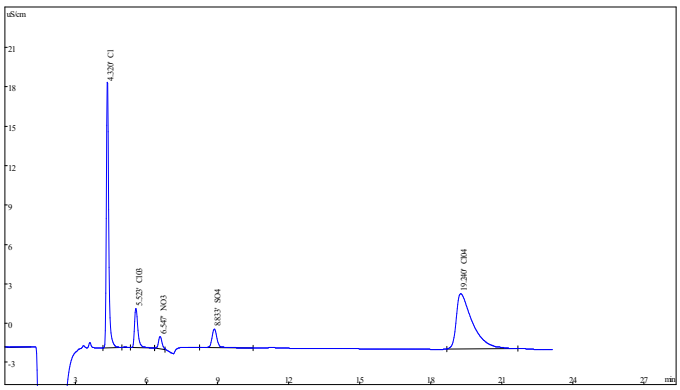

Figure 4. Sample added scalar quantity0.1mg/kg

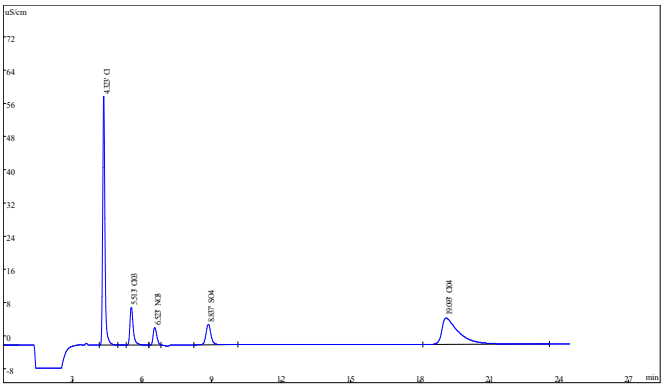

Figure 5. Sample added scalar quantity0.5mg/kg

Results and calculations

Table 7: Sample test result

Ions	added scalar quantity mg/kg	Concentration mg/L	Recycling rates %
Cl^-	0	0.1329	--
	0.05	0.1821	98.40
	0.1	0.2267	93.80
	0.5	0.6395	101.3
ClO_3^-	0	0.0829	--
	0.04	0.1238	102.3
	0.08	0.1593	95.50
	0.4	0.4724	97.38
NO_3^-	0	0.003009	--
	0.02	0.02221	96.01
	0.04	0.03931	90.75
	0.2	0.1895	93.25
SO_4^{2-}	0	0.03291	--
	0.02	0.04225	93.40
	0.04	0.05213	96.10
	0.2	0.1991	92.33
ClO_4^-	0	1.174	--
	0.5	1.687	102.6
	1	2.225	105.1
	2	3.445	113.6

Remarks: ① ND indicates not detected; ② The measured value has been deducted from the blank value;

③ 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.

After testing, under chromatograph conditions, the five anions in explosives can be separated within 20 minutes, and the pre-treatment method is simple. Under these conditions, the stability of the tested ions is good, and the linear correlation coefficients can all be above 0.999, which meets the testing requirements and can be applied to the testing of fire and explosives in the public security industry.