Determination of fluoride and chloride in coal tar

Introduction:

Chloride in coal tar is divided into inorganic chlorides and organic chlorides. Sodium chloride in inorganic chlorides is not easy to be hydrolyzed, but magnesium chloride and calcium chloride are very easy to be hydrolyzed by heat. Their hydrolysis temperatures are 120℃ and 175℃ respectively. After hydrolysis, hydrogen chloride is generated as a corrosive medium. When organic chlorides exist alone, they do not cause corrosion to equipment. However, under conditions of high temperature, high pressure, and the presence of hydrogen gas, hydrogen chloride is generated. When water is present, it has strong corrosiveness to equipment. In addition, due to the unbound lone pair electrons and great electron affinity of fluorine and chlorine, it is easy to react with metal ions, and the chloride ions also have high mobility, which often migrate downstream with the process, resulting in full bed catalyst poisoning.

Detection items (Table 1):

Anion	F^-	Cl^-

Keywords: Ion chromatography, Coal tar,Fluoride, Chloride

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.F^-、Cl^- 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:

Instrument	CIC-D160⁺
Eluent	0-15 min，5.0 mMKOH 15.1-30 min，35.0 mMKOH 30.1-35 min，5.0 mMKOH
Flow rate	1.0 mL/min
Injection volume	25 μL
Analytical Column	AG19+AS19
Column oven temperature	30℃
Conductivity cell temperature	35℃
Suppressor current	90 mA

Sample preparation

Weigh an appropriate amount of sample, place it in a crucible, burn and absorb it with an oxygen bomb, and pass through 0.22 μ After filtering with a membrane, analyze the sample.

Table 4:Sample preparation table

Sample	Weight（g）	Volume(mL)	Ions
1#	0.1000	100.0	F^-、Cl^-
2#	0.1949	100.0	F^-、Cl^-

Standard chromatogram

Standard chromatogram, As shown in below:

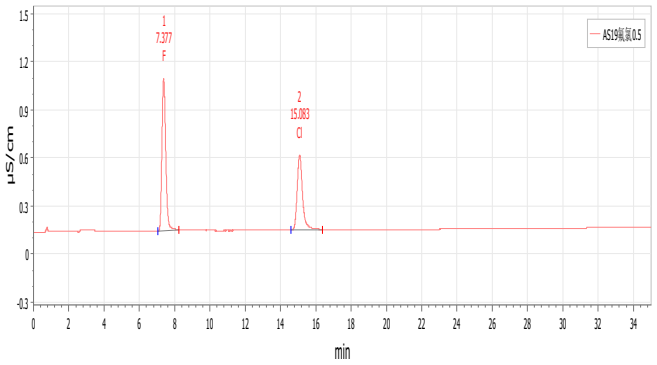

Figure 1. Chromatogram of standard sample.

Table 5 :Standard sample data

Ions	Time [min]	Concentration [mg/L]	Area [(μS/cm)*min]
F^-	7.376667	0.5000	0.240493
Cl^-	15.083333	0.5000	0.165555

Blank chromatogram

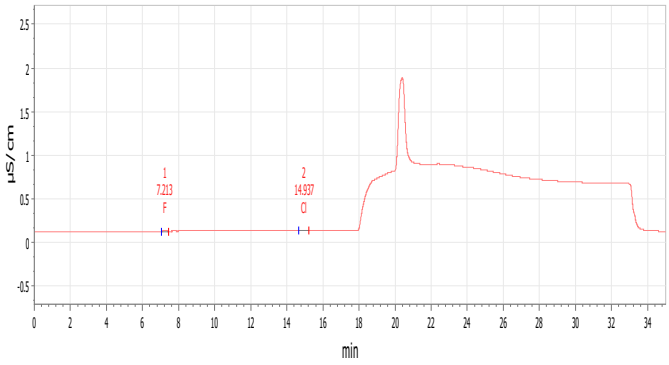

Figure 2. Chromatogram of blank

Sample chromatogram

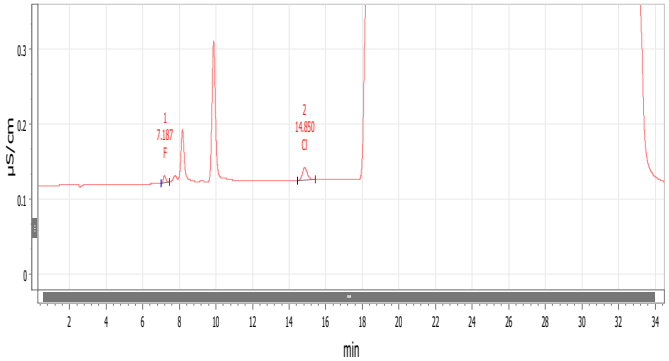

Figure 3. Chromatogram of sample 1#

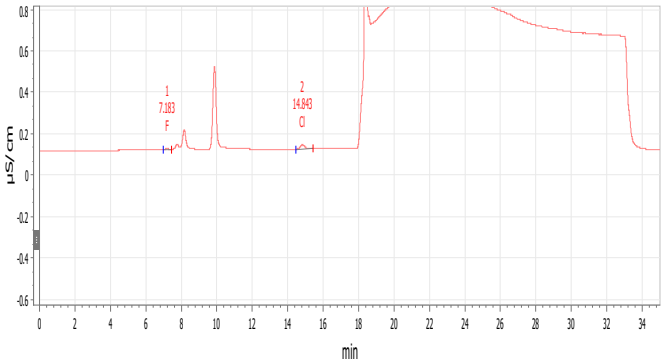

Figure 4. Chromatogram of sample 2#

Results and calculations

Table 6: Sample test result (mg/kg)

Ions	F^-	Cl^-
Sample 1#	≤5 mg/kg	≤10 mg/kg
Sample 2#	≤5 mg/kg	≤10 mg/kg

Remarks: ① Blank space has been deducted from the test results; 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.