Detection of chlorate and perchlorate in milk

Introduction:

Chlorate is a byproduct produced during the disinfection process. If chlorine containing disinfectants are used in milk production, it may lead to excessive levels of chlorate. Secondly, chlorine containing cleaning agents used in milking and milk processing plants may also be sources of pollution. In addition, if animal feed contains perchlorates, these substances may enter milk.

Perchlorate has certain hazards to the human body, such as hindering the absorption of iodine by the thyroid gland, damaging red blood cells, and affecting the function of blood in transporting oxygen. This article uses ion chromatography to determine the content of chlorate and perchlorate in milk.

Detection items (Table 1):

Anion	ClO_3^-	ClO_4^-

Keywords: Chlorate，Perchlorate，Milk， Ion chromatography

Instruments and equipment

- **Ion chromatograph**: CIC-D180
- **Ultra pure water machine**:ECO-S15

Qingdao Shenghan Chromatograph Technology Co., Ltd

Requirements

Reagents

Unless otherwise specified, all reagents used are superior grade. Commercially available certified standard solutions for ClO_3^- ,ClO_4^- (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-D180
Eluent	0-14 min 8 mM KOH 14.1-45 min 35 mM KOH 45.1-50 min 8 mM KOH
Flow rate	1.0 mL/min
Injection volume	500 μL
Analytical Column	IonPac AG19+IonPac AS19
Column oven temperature	30℃
Conductivity cell temperature	35℃
Suppressor current	100 mA

Sample preparation

Weigh about 2 mL of milk sample, add 2 mL of acetonitrile, and flocculent precipitate will be found. Then add 4 mL of ultrapure water, vortex for 30 seconds, centrifuge at 8000 r/min for 5 minutes, take 1 mL of supernatant and make up to 10 mL, vortex well,pass through activate C18 pre-treatment column with 0.22 μm filter membrane, and perform ion chromatography test.

Standard chromatogram

Standard chromatogram, As shown in below:

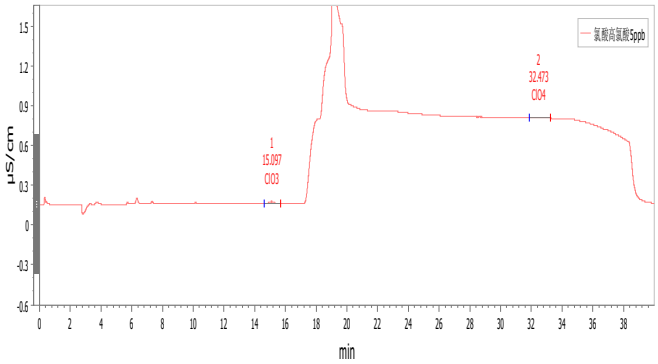

Figure 1. Chromatogram of standard sample.

Table 4: Standard sample result

Ions	Time[min]	c[mg/L]	Area[(μS/cm)*min]
$ClO3^-$	15.096667	0.005000	0.004547
$ClO4^-$	32.473333	0.005000	0.003449

Blank chromatogram

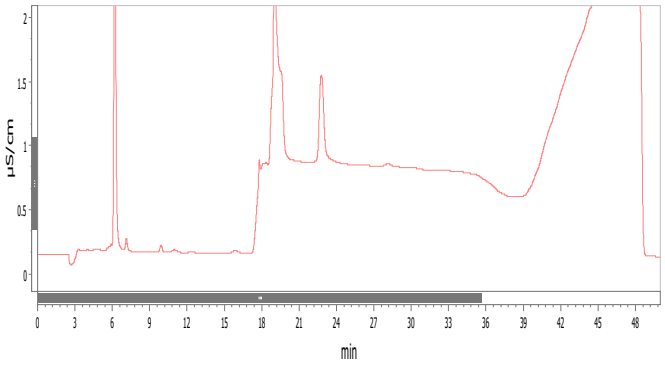

Figure 2. Chromatogram of blank

Sample chromatogram

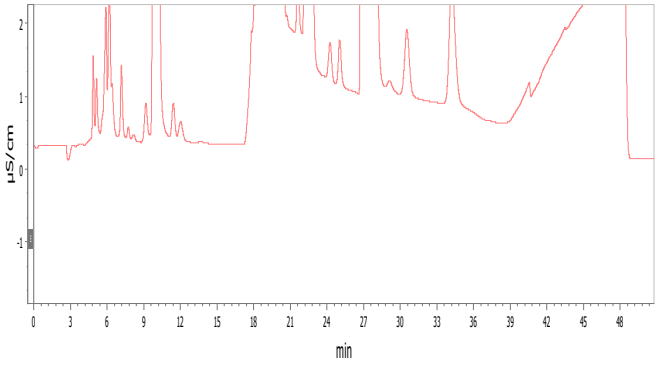

Figure 3. Chromatogram of sample

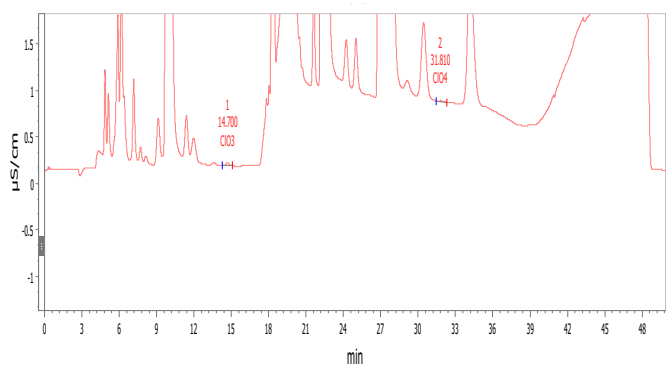

Figure 4. Chromatogram of sample add standard

Results and calculations

Table 5: Sample test result (unit: mg/L)

Sample	ClO_3^-	ClO_4^-
1	ND	ND

Remarks: ① ND indicates that the result was not detected or was below the detection limit. ② There may be differences in testing results between different methods and laboratories.

Feasibility analysis and conclusion

Through the above experiments, it has been proven that the detection method has good separation and is suitable for the determination of the content of the components to be tested in the sample. After testing, the detection limit of the method for chlorates and perchlorates in actual sample milk is 0.5 mg/kg.