# Application solution of ion chromatography in food analysis and determination

various phosphate

#### Foreword

Phosphate is a widely used food additive and plays an important role in improving food quality. At present, food phosphates mainly include sodium salt, potassium salt, calcium salt, iron salt, zinc salt and so on. Phosphate is mainly used as water retainer, bulking agent, acidity regulator, stabilizer, coagulant and potassium ferrocyanide in food. Current national standard GB 2760-2014 "national food safety standard-Standards for the use of food additives" clearly points out the types of phosphate additives that can be used in food and the maximum use requirements. A total of 19 kinds of phosphate are allowed to use.

Amongthem,trisodiumphosphateanhydrous,sodiumhexametaphosphate,sodiumpyrophosphate,sodiumTripolyphosphate,sodium trimetaphosphate and so on can be added intothe specified food types in accordance with the specified amount.Calciumhydrogen phosphate and sodium dihydrogen phosphate are only used ininfant formula food and infant supplementary food,and The maximumdosage of single or mixed use is 1.0g/kg with PO43-.

## **Implementation standard**

The determination methods of various phosphates in foods are



specified in National standard GB5009.256-2016 "national food safety standard-the determination of various phosphate in food". It is specified that ion chromatography combined with gradient elution is used for the determination of phosphate, pyrophosphate, hexametaphosphate, sodium trimetaphosphate, sodium Tripolyphosphate in food in the standard.

It is also pointed out that ion chromatography can be used for the analysis and determination of various phosphates in Entry-Exit Inspection and Quarantine Industry Standard SN/T 4590-2016 "The content determination of pyrophosphate, sodium Tripolyphosphate,trimetaphosphate in export aquatic products by ion chromatography".

## **Reagents and standards**

1.Sodium hydroxide(NaOH),GR

2.Methanol(CH<sub>3</sub>OH), chromatographic pure

3.Trisodiumphosphateanhydrous(Na<sub>3</sub>PO<sub>4</sub>)standardsolution(1000mg/L,water matrix)

4.Sodium pyrophosphate(Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>)standard solution(1000mg/L,water matrix)

5.Sodium trimetaphosphate[(NaPO<sub>3</sub>)<sub>3</sub>]standard:purity ≥98%

6.Sodium Tripolyphosphate(Na<sub>5</sub>P<sub>3</sub>O<sub>10</sub>)standard:purity  $\geq$  98%

Configuration and chromatographic conditions:



- IC type:CIC-D160(Built-in eluent generator)
- IC column:SH-AC-16(Analysis column for polyphosphate)
- Guard column:SH-G-1
- Eluent:KOH gradient elution
- Flow rate:1.0mL/min
- Sample size:25  $\mu$  L
- Detection method: Suppressed conductivity method

#### • Pretreatment:C18 column, H column ,Na column

| Time(min) | Concentration(mM) |
|-----------|-------------------|
| 0.0-9.0   | 30.0              |
| 9.1-11.0  | 30.0-50.0         |
| 11.1-28.0 | 50.0              |
| 28.1-31.0 | 30.0              |
| 30.0-40.0 | 30.0              |

Table1 Gradient elution concentration settings reference

## Pretreatment

Sample pretreatment refers to the method which is specified in National standard GB5009.256-2016 "national food safety standard-the determination of various phosphate in food".

## **Test spectrum**





Table1 Spectrogram of mixed sample of phosphate radical, Pyrophosphoric acid radical, Tripolyphosphate radical and Trimetaphosphoric acid radical

| No.           | Phosphate | Pyrophosphoric | Tripolyphosph | Trimetaphosph |
|---------------|-----------|----------------|---------------|---------------|
|               | radical   | acid radical   | ate radical   | oric acid     |
|               |           |                |               | radical       |
| 1             | 9.348     | 16.004         | 17.713        | 21.156        |
| 2             | 9.391     | 16.035         | 17.745        | 21.18         |
| 3             | 9.403     | 16.025         | 17.708        | 21.133        |
| 4             | 9.398     | 16.03          | 17.735        | 21.12         |
| 5             | 9.412     | 16.028         | 17.726        | 21.126        |
| 6             | 9.409     | 16.037         | 17.765        | 21.139        |
| Average value | 9.394     | 16.027         | 17.732        | 21.142        |
| Standard      | 0.024     | 0.012          | 0.021         | 0.022         |
| deviations    |           |                |               |               |
| Relative      | 0.251     | 0.074          | 0.119         | 0.105         |
| standard      |           |                |               |               |
| deviation%    |           |                |               |               |

# **Qualitative repeatablity**

# Quantitative repeatability

| No. | Phosphate | Pyrophosphoric | Tripolyphosph | Trimetaphosph |
|-----|-----------|----------------|---------------|---------------|
|     | radical   | acid radical   | ate radical   | oric acid     |
|     |           |                |               | radical       |
| 1   | 2223745   | 8304960        | 10872762      | 3421433       |



| 2             | 2239273    | 8335228    | 10890442    | 3413376    |
|---------------|------------|------------|-------------|------------|
| 3             | 2231097    | 8272525    | 10840304    | 3392714    |
| 4             | 2232084    | 8306597    | 10853267    | 3400126    |
| 5             | 2233006    | 8308989    | 10859631    | 3401089    |
| 6             | 2233098    | 8310067    | 10856629    | 3401563    |
| Average value | 2232050.50 | 8306394.33 | 10862172.50 | 3405050.17 |
| Standard      | 4981.29    | 20004.16   | 17355.29    | 10411.50   |
| deviations    |            |            |             |            |
| Relative      | 0.22       | 0.24       | 0.16        | 0.31       |
| standard      |            |            |             |            |
| deviation%    |            |            |             |            |

## Conclusion

In this paper, ion chromatographic separation and combined with KOH gradient elution technique were used to separate perfectly phosphate radical,pyrophosphoric acid radical,tripolyphosphate radical,and trimetaphosphoric acid radical and can be used for qualitative and quantitative analysis of four phosphoric acid compounds.The method has the advantages of simple operation, high sensitivity, high precision and accuracy, and can be used for the determination of various phosphates in food.



## **Product presentation**



## CIC-D160 IC

CIC-D160 ion chromatograph is the first hydrogen-oxygen ion chromatograph made in China. It is equipped with bipolar conductance detector which greatly improves the detection ability, stability of the instrument, and brings the best usage experience to the users. Its built-in eluent generator can generate the required concentration of eluent on line by pure water and possesses the function of gradient elution which can determines complex samples which isocratic elution cannot. Now It is widely used in the environment, food, chemical industry, power, electronics, mining and metallurgy and other fields.

■Built-in eluent generator:No need to configure the eluent and possesses the function of gradient elution;

Built-in circulating 3D constant temperature technology which ensure the accuracy and reliability of the experimental data;

■Built-in low-pressure degassing technology to eliminate bubble interference for more stability;

Self-regenerating electrolytic micro-membrane suppressor which pressure resistance is high, dead volume is small, and responsive signal is high;

Equipped with intelligent automatic injection system for large sample volumes, which features automatic dilution to save labor and time;

•Observatory intelligent workstation which is configured with integrated control, compatibility for a variety of instruments, and customized images.

■ Perfect after-sale support to solve the worries of users

## Ion Chromatographic Column





AS the first domestic developer and manufacturer of Ion chromatographic colum, Sheng Han have the technology of the development and production of three kinds of Ion chromatographic column including ion exchange chromatographic column, ion exclusion chromatographic column and ion pair chromatographic column. At the same time, Sheng Han have also successfully developed and produced hydroxyl system of Ion chromatographic column in large scale ranking second in the world, which broken the monopoly of imported brands in the high-end ion chromatographic column field more than ten years. The use of domestic ion chromatography can reduce the cost of operation and maintenance of users by about 35%.

