SHINE 盛瀚

ION CHROMATOGRAPHY APPLICATION NOTES

Food Industry

CATALOGUE

1.1 Determination of nitrate in soybean powder

Analysis Conditions:

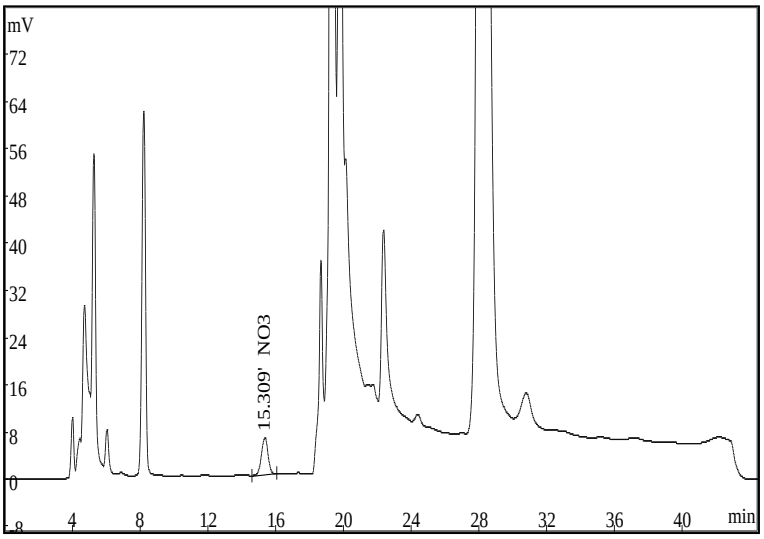
- Analytical Column：SH-G-1+SH-AC-11
- Mobile Phase：KOH Gradient elution

  0-15 min, 10 mM KOH;

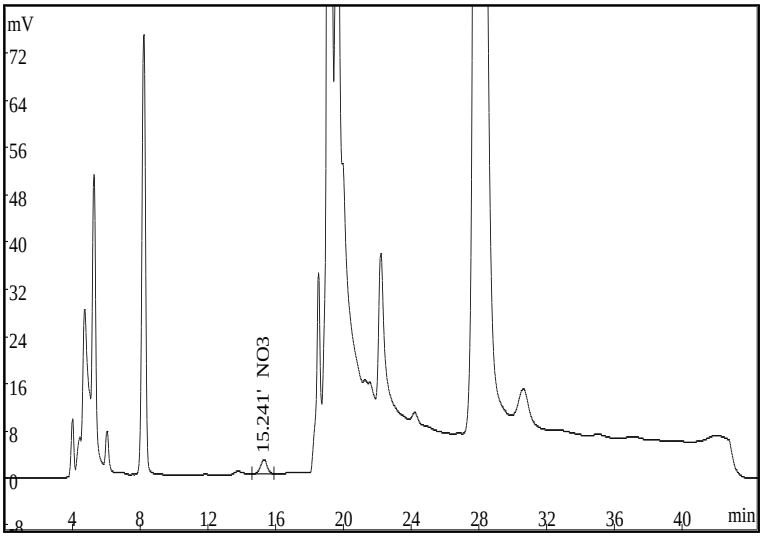
  15.1-40 min, 30 mM KOH;

  40-45 min, 10 mM KOH.
- Flow Rate：1.0 mL/min
- Injection Volume：25 μL
- Suppressor：SHY-A-6

Pre-treatment: Weigh a proper amount of sample and put it into a 10 ml volumetric flask, add 5 ml deionized water to mix well, and extract it by ultrasonic for 30 min. Shake well with acetonitrile at a constant volume and allow to stand for 10 minutes. After centrifugation for 20 min at 10000 R / min, the supernatant was diluted 10 times, and analyzed by C18 column and 0.22 μ m disposable needle filter.



Chromatogram of nitrate in bean powder 03



Chromatogram of nitrate in bean powder 25

1.2 Determination of nitrite and nitrate in fructooligosaccharide and beetroot

Analysis Conditions:

- Analytical Column：SH-G-1+SH-AC-11
- Mobile Phase：KOH Gradient elution

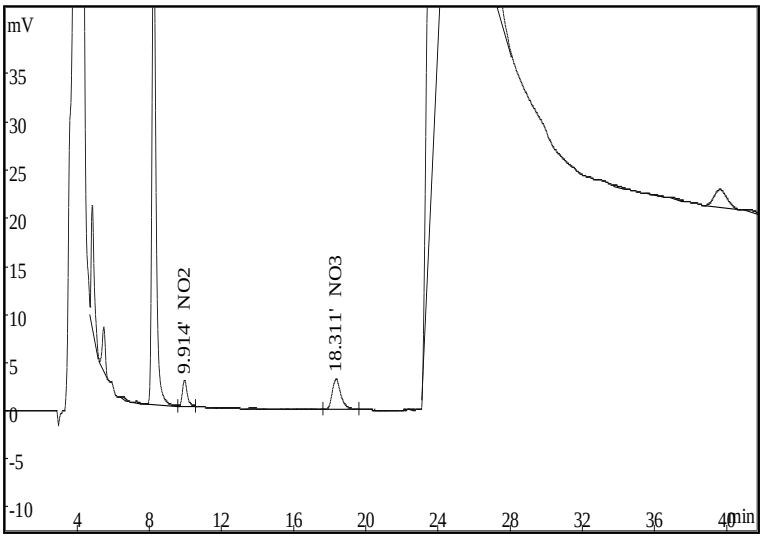
  0-20 min, 6 mM KOH;

  20.1-45 min, 50mM KOH;

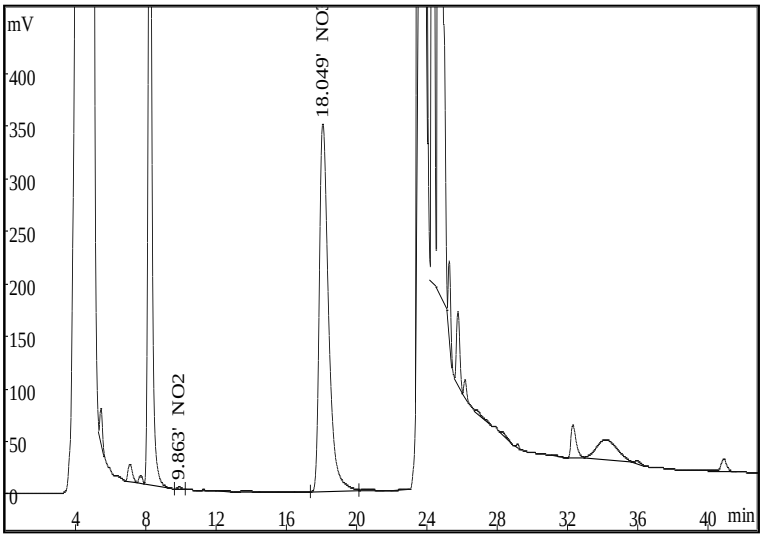
  45-50 min, 6 mM KOH.
- Flow Rate：1.0 mL/min
- Injection Volume：50 µL
- Suppressor：SHY-A-6

Pre-treatment：Wash and dry the beetroot, cut it into small pieces, and then put it into the blender. Accurately weigh 5.0 g of the broken sample into a 150 ml conical flask with a plug, add 80 ml of deionized water and 1 ml of 1mol/L NaOH solution, and conduct ultrasound for 30 min. Then put it into a 75 ℃ water bath for 5 min, take it out and cool it to room temperature, transfer it to a 100 ml volumetric flask, wash the conical flask with deionized water, put the lotion into a 100 ml volumetric flask, and shake it evenly at a constant volume. After centrifugation for 15 min at

10000 R / min, the supernatant was taken to pass through 0.22 μ m disposable needle filter and C18 pre-treatment column for sample injection.



Chromatogram of nitrite and nitrate in oligofructose



Chromatogram of nitrite and nitrate in beet root

1.3 Determination of sodium and calcium in soybean powder

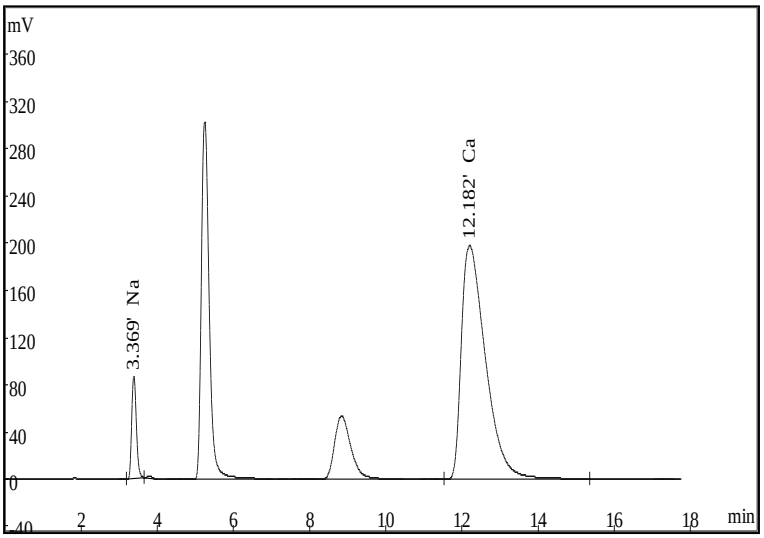
Nutritional enhancer refers to the natural or synthetic nutrients and other nutrients added to the food in order to increase the nutritional components of the food. The purpose is mainly to make up for the nutritional loss of the food in the process of processing and storage, and improve the health impact caused by low or lack of intake level through strengthening. Gb14880-2012 specifies the application range and amount of nutritional fortifier in food, among which, the amount of calcium in soybean powder is 1600-8000 mg / kg.

Analysis Conditions:

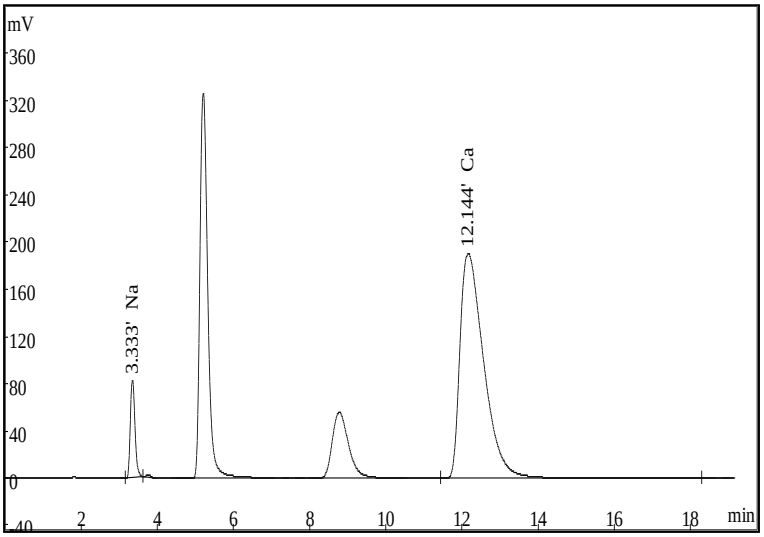
- Analytical Column: SH-G-1+SH-CC-4
- Mobile Phase：5.0 mM MSA
- Flow Rate：1.0 mL/min
- Suppressor：SHY-C-3
- Injection Volume：25 μL

Pre-treatment: accurately weigh a proper amount of sample and put it into a 30ml

porcelain crucible, put it in a heating sleeve and heat it to carbonize it at 200 ℃ until it does not smoke. Transfer to muffle furnace for roasting at 550 ℃, take it out and put it at room temperature after cooling. Dissolve the solid with nitric acid solution, transfer it to a 100ml volumetric flask, wash the crucible with deionized water, and put the washing solution into the volumetric flask, and finally shake it with deionized water at a constant volume. After diluting 10 times, sample was injected through 0.22 μ m disposable needle filter for analysis.



Chromatogram of sodium and calcium in soybean flour 03



Chromatogram of sodium and calcium in soybean flour 25

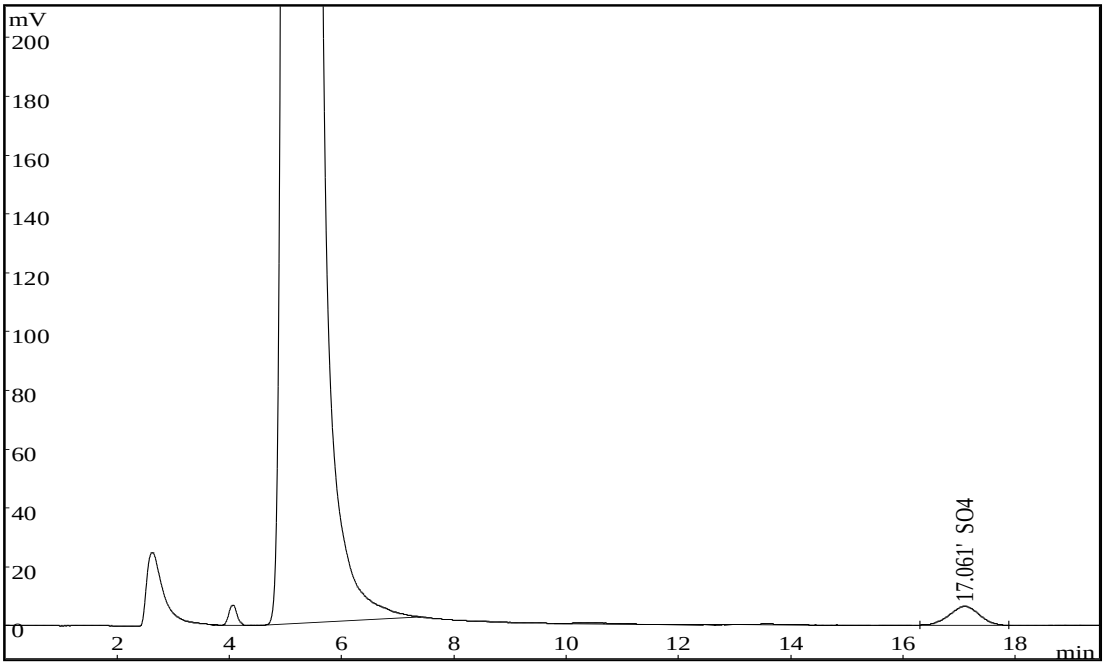
1.4 Determination of sulfate ion in cyclamate

Sodium cyclamate, whose chemical name is sodium cyclamate, is a common additive in food production. In the quality standard of sodium cyclamate, there is a clear requirement for the content of sulfate, which is ≤ 0.05%. The content of sulfate ion in cyclamate can be determined by ion chromatography.

Analysis Conditions:

- Analytical Column：SH-G-1+SH-AC-4
- Mobile Phase：2.0 mM Na2CO3+8.0 mM NaHCO3
- Flow Rate：1.3 mL/min
- Suppressor：SHY-A-6
- Injection Volume：25 μL

Pre-treatment:：Weigh about 0.5g of sample, accurately record the mass, accurate to 0.0001 g, dilute to 100 ml with ultra pure water, and inject the sample through 0.22 μ M filter membrane for analysis.



Chromatogram of sulfate ion in sodium cyclamate

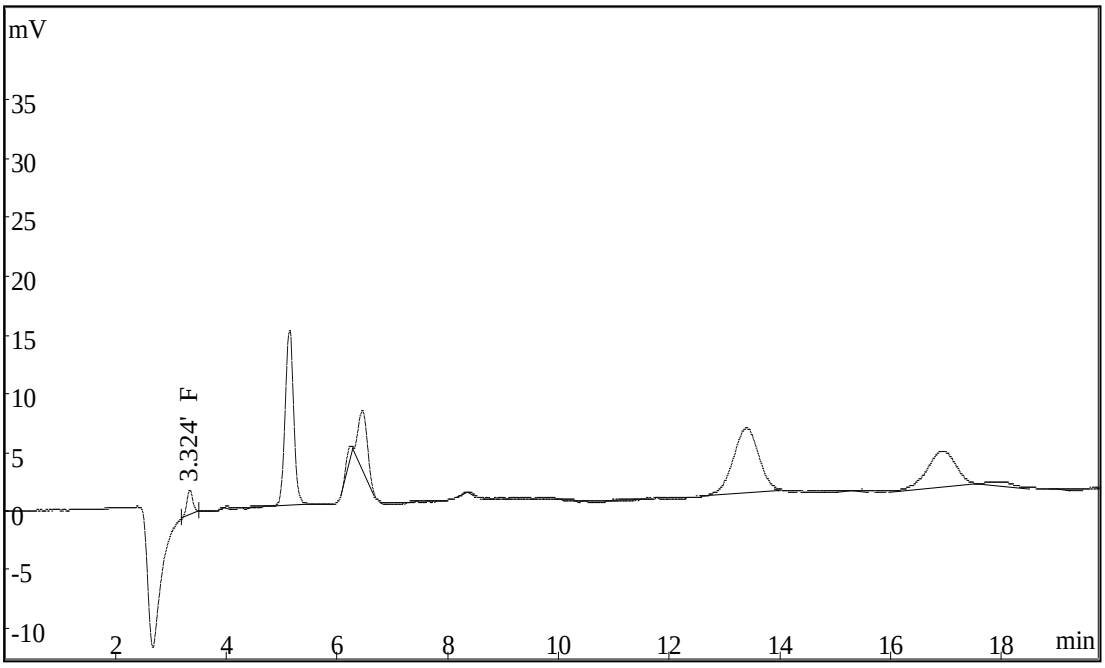
1.5 Determination of fluorine in krill oil

The protein content of krill is more than 50%, and it is also rich in amino acids and vitamin A which are essential for human tissues. Prawn oil contains omega-3 essential fatty acids, eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), which can promote the protection of cardiovascular system, nerves, bone joints and improve human immunity.

Analysis Conditions:

- Analytical Column：SH-G-1+SH-AC-4
- Mobile Phase：2.0 mM Na_2CO_3+8.0 mM $NaHCO_3$
- Flow Rate：1.3 mL/min
- Suppressor：SHY-A-6
- Injection Volume：25 µL

Pre-treatment：Accurately weigh 20-30 mg sample (accurate to 0.0001 g), add it into sample boat, and analyze fluorine in sample by on-line combustion ion chromatography.



Fluorine chromatogram of krill oil

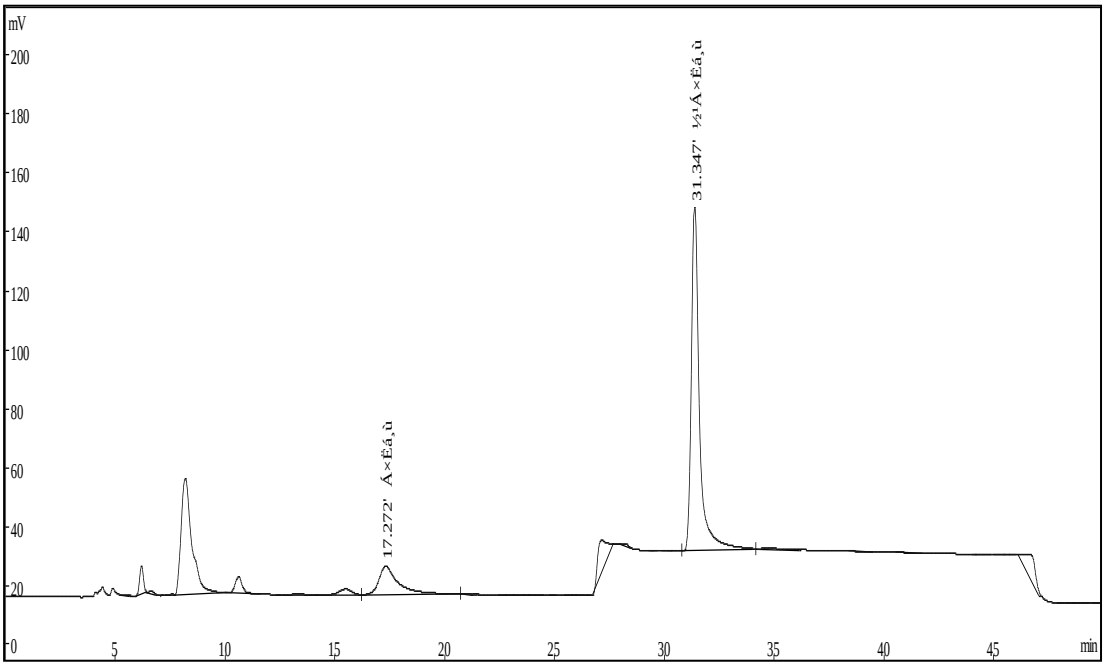
1.6 Determination of phosphate and pyrophosphate in fish meat

Polyphosphate is a kind of widely used food additive. As an important food ingredient and functional additive, polyphosphate is widely used in the processing of meat products, seafood, seasoning, convenience food, etc. It plays an important role in the improvement of food quality. It is mainly used as water retention agent to improve the water retention of the product and keep the meat tender. However, excessive intake of polyphosphate will have certain harm to the body. Gb2760-2011 specifies the use range and amount of polyphosphate in food, among which the use limit of polyphosphate (calculated by phosphate radical) in prefabricated aquatic products is 1.0 g / kg. There are also clear regulations on the use of polyphosphate in food in foreign countries, among which the European Union requires that the content of polyphosphate in aquatic products shall not exceed 0.5g/kg.

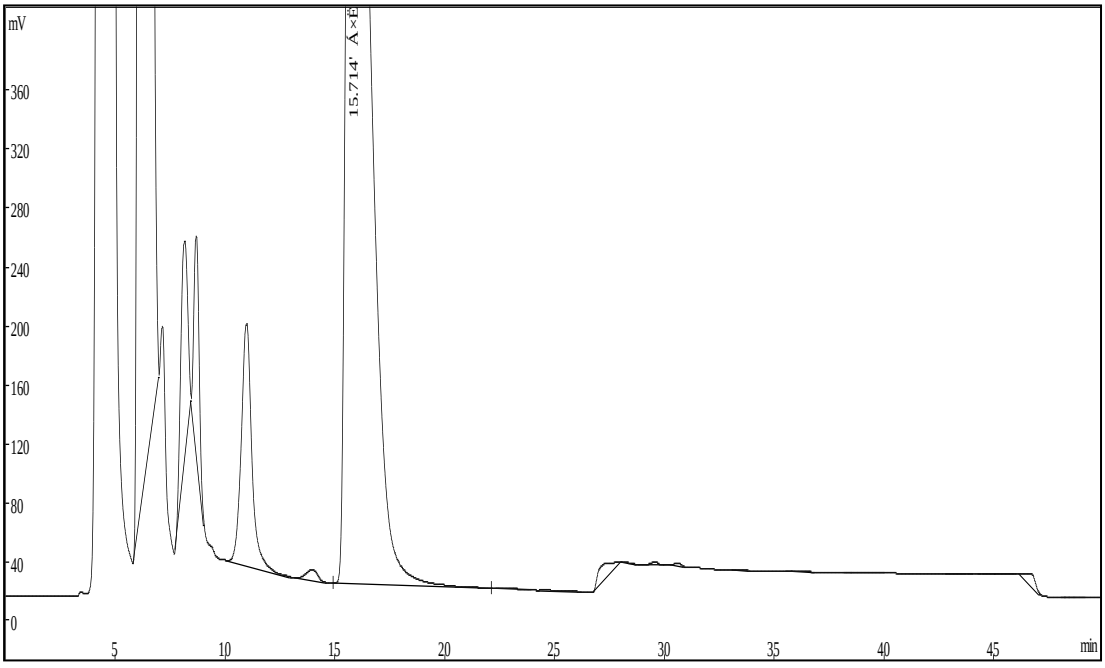
Analysis Conditions:

- Analytical Column：SH-G-1+SH-AC-16
- Mobile Phase：KOH Gradient Eluent
- 0-23 min: 25 mM KOH; 23.1-45 min: 45 mM KOH; 45.1-50 min: 25 mM KOH.
- Flow Rate：1.0 mL/min
- Suppressor：SHY-A-6
- Injection Volume：25 μL

Pre-treatment：Accurately weigh a proper amount of fish sample (accurate to 0.1 mg), wash it with 50 mmol / L sodium hydroxide solution into 100 ml colorimetric tube and fix the volume. Ultrasonic extraction at 80 ℃ for 30 minutes, shaking every 5 minutes to keep the solid phase completely dispersed. After cooling to room temperature, the filter paper is filtered and the filtrate is centrifuged at 10000 R / min for 10 min. After diluting the supernatant in a proper multiple, the supernatant was analyzed by 0.22 μ m disposable needle filter and C18 injection.



Chromatogram of phosphate and pyrophosphate in fish



Chromatogram of phosphate and pyrophosphate in rotten fish

1.7 Determination of nitrite and nitrate ion in fatty acid special medical

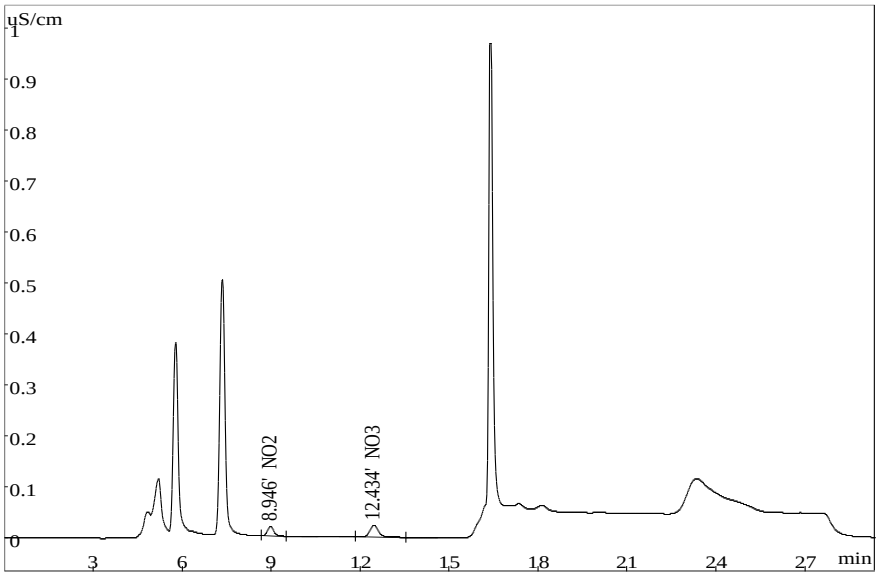
food

Food for special medical purpose (fsmp), referred to as medical food, is specially processed and formulated to meet the special needs of people with eating restriction, digestion and absorption disorder, metabolic disorder or specific disease status for nutrients or diet. Nitrate and nitrite, as food preservatives and preservatives, can prolong the shelf life, but have toxic effects on human body. Therefore, monitoring the use of nitrate and nitrite in medical food is of great significance.

Analysis Conditions:

- Analytical Column：SH-G-1+SH-AC-11
- Mobile Phase：KOH Gradient elution
- Gradient condition: 0-12 min: 13 mM KOH; 12.1-24 min: 30 mM KOH; 24.1-30 min: 13 mM KOH.
- Flow Rate：1.0 mL/min
- Column Temperature：35℃
- Injection Volume：50 µL
- Suppressor：SHY-A-6
- Pretreatment: weigh 5.0 g sample (accurately record the mass, accurate to 0.0001 g), put it into a 150 ml conical flask with stopper, add 80 ml deionized

water, and extract with ultrasonic for 30 min. Shake once every 5 minutes to keep the solid phase completely dispersed. Put it in a 75 ℃ water bath for 5 min, take it out and place it at room temperature, transfer the quantitative amount to a 100 ml volumetric flask, wash the conical flask with deionized water, and merge the lotion into a 100 ml volumetric flask, and shake up at constant volume. After the solution was filtered by filter paper, part of the solution was centrifuged at 10000 R / min for 15 min. the supernatant was passed through a 0.22 μ m disposable needle filter and a C18 pretreatment column for sample injection.



Chromatogram of nitrite and nitrate ion in fatty acid special medical food

1.8 Determination of bromate in wheat flour

In the 21st century, with the continuous improvement of people's living standards, food additives are required to be safe, efficient and broad-spectrum, which requires that the selection of flour additives should consider their safety while determining their high efficiency. For a long time, potassium bromate has been an important member of flour additive family, which has a special and efficient effect on flour improvement. However, its carcinogenicity has been widely confirmed, potassium bromate can cause chronic poisoning and lead to a variety of diseases; short-term excessive consumption will cause nausea, dizziness, neurasthenia and other toxic symptoms, and the harm to the human body is obvious.

Analysis Conditions:

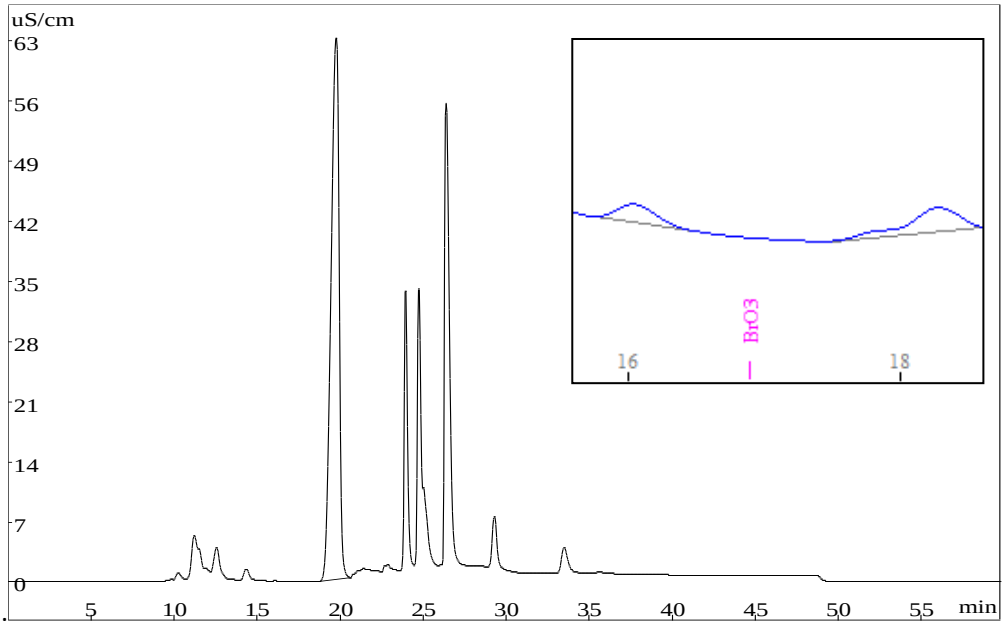
- Analytical Column：SH-G-1+SH-AP-1
- Mobile Phase：KOH Gradient Eluent
- Gradient condition：0-17 min: 5 mM KOH; 17.1-45 min: 30 mM KOH; 45.1-60 min: 5 mM KOH.
- Flow Rate：0.7 mL/min

- Column Temperature：45℃
- Injection Volume：25 μL
- Suppressor：SHY-A-6
- Pretreatment：
- (1) Accurately weigh 10 g (accurate to 0.1 g) of wheat flour into a 250 ml triangular flask with stopper, add 100.0 ml of ultra pure water, shake up quickly, put an oscillator for 20 min (extract 20 min in ultrasonic under intermittent stirring), stand still, transfer 20 ml of upper liquid into a 50 ml centrifuge tube, 3000 R / min away from the center for 20 min, and the supernatant is standby;

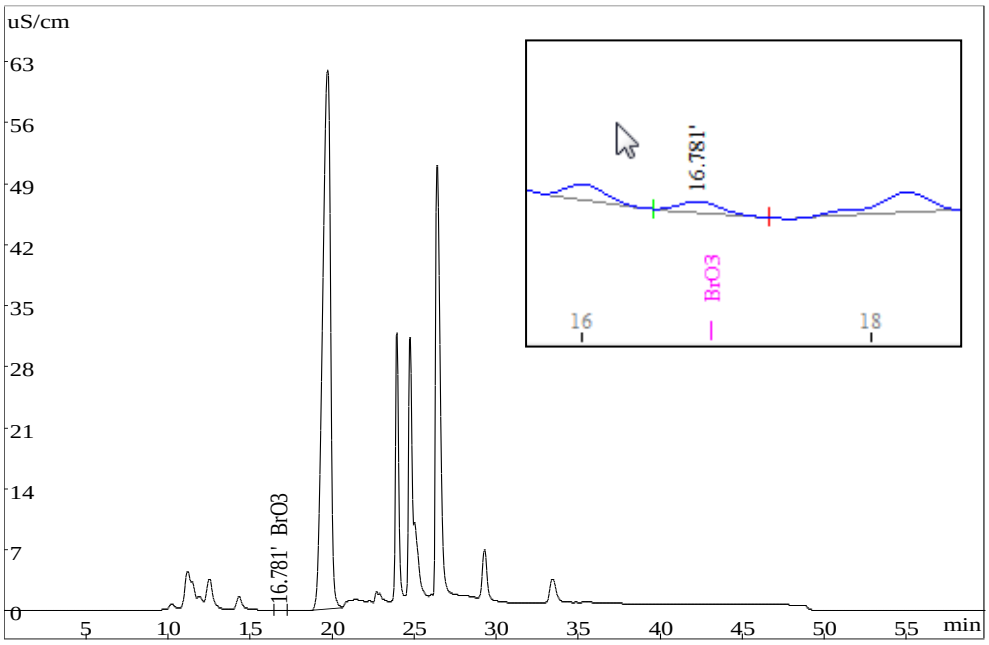
  (2) The supernatant was passed through the Ag / h column to remove Cl - in the sample extraction solution, and the operation was carried out according to the product instructions. For wheat flour with Cl - content less than 1 g / kg, this operation can also be omitted;

  (3) The sample extract was filtered by 0.22 μ m membrane and then injected into the ultrafiltration sample cup. Centrifugation was performed at 10 000 R / min for 30 min for ultrafiltration. The ultrafiltration filtrate was directly analyzed by chromatography.

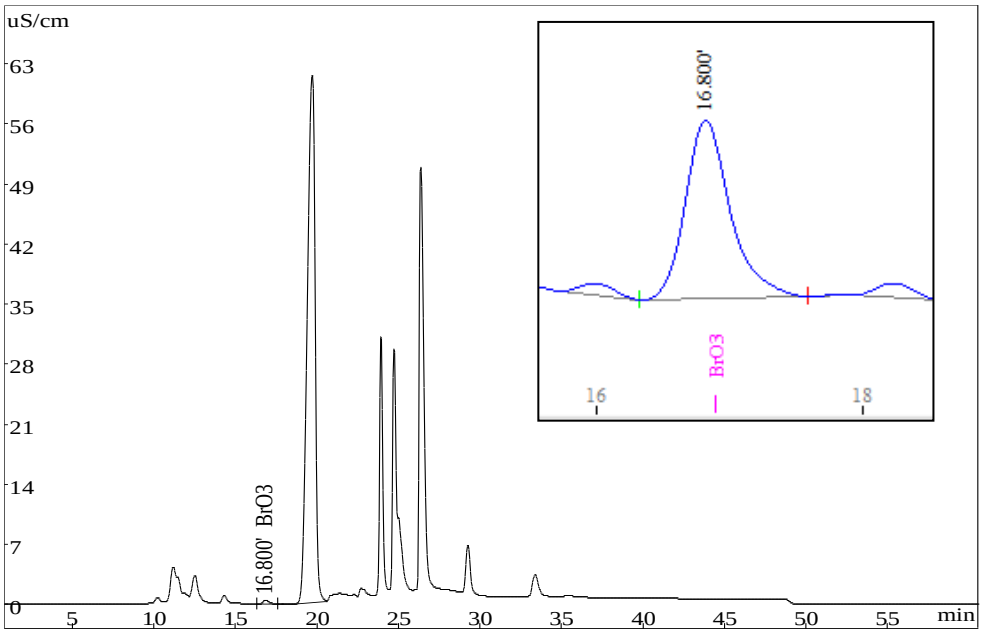
  (4) 10 g wheat flour was added with 1 mg / kg BrO3 - and 20 mg / kg BrO3 - respectively.



Blank chromatoram of wheat flour



Chromatogram of wheat flour spiked with 1 mg / kg BrO3



Chromatogram of wheat flour spiked with 20 mg / kg BrO3

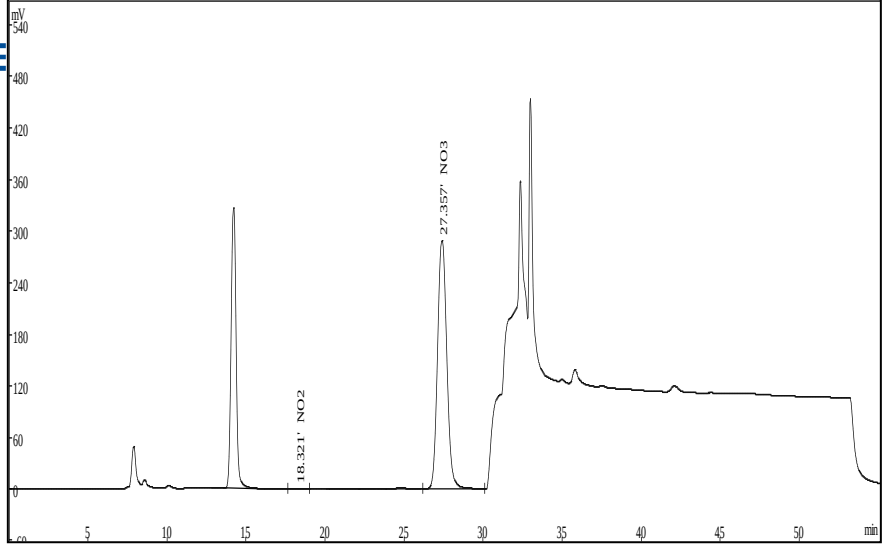
1.9 Determination of nitrite and nitrate ion in vegetable powder

Vegetable powder is a powder vegetable granule formed from drying and dehydration of vegetable raw materials, then further crushing, or first beating, then

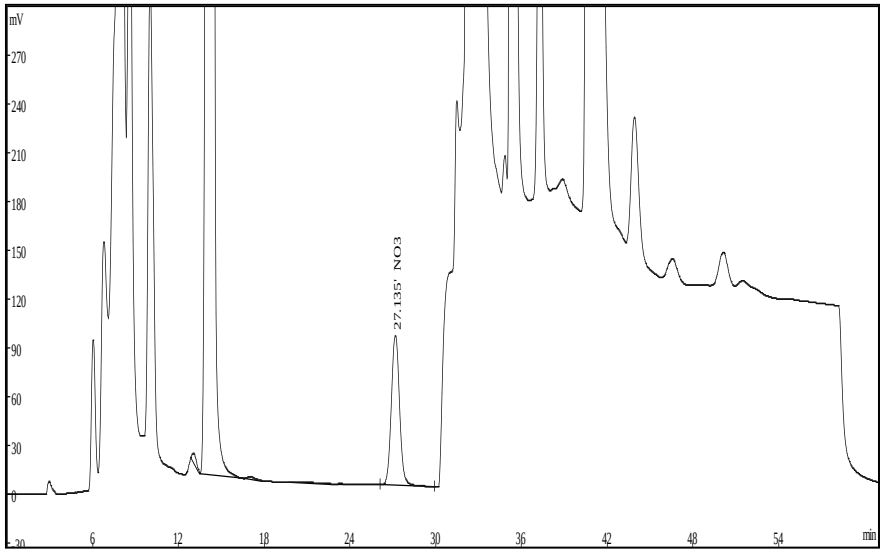
spray drying. Vegetable powder is an extended product of dehydrated vegetables, which has a wide market in infants, the elderly and other people. In addition, vegetable powder is also a natural colorant, which is an excellent raw material for processing vegetable bread, vegetable noodles and special food. Nitrate and nitrite, as food preservatives and preservatives, can prolong the shelf life, but have toxic effects on human body. Therefore, it is of great significance to monitor the amount of nitrate and nitrite in vegetable powder.

Analysis Conditions:

- Analytical Column：SH-G-1+SH-AC-11
- Mobile Phase：KOH Gradient Eluent
- Gradient condition：0-28 min: 5 mM KOH; 28.1-55 min: 30 mM KOH; 55.1-60 min: 5 mM KOH.
- Flow Rate：1.0 mL/min
- Column Temperature：35℃
- Injection Volume：25 μL
- Suppressor：SHY-A-6
- Pretreatment：Accurately weigh 5.0 g sample (accurate to 0.1 mg) into a 150 ml triangular flask with stopper, add 80 ml high-purity water and 1.0 ml1.0 mol / L sodium hydroxide solution, shake for 20 min, and ultrasonic extraction for 30 min. Water bath at 75 ℃ for 5 min, take out and cool to room temperature. Transfer to a 100 ml volumetric flask and shake with constant volume. The supernatant was centrifuged at 12000 R / min for 20 min, and the supernatant was diluted according to the content. The supernatant was passed through 0.22 μ m disposable needle filter and C18 column for sample injection analysis.



Chromatogram of nitrite and nitrate ion in white radish powder



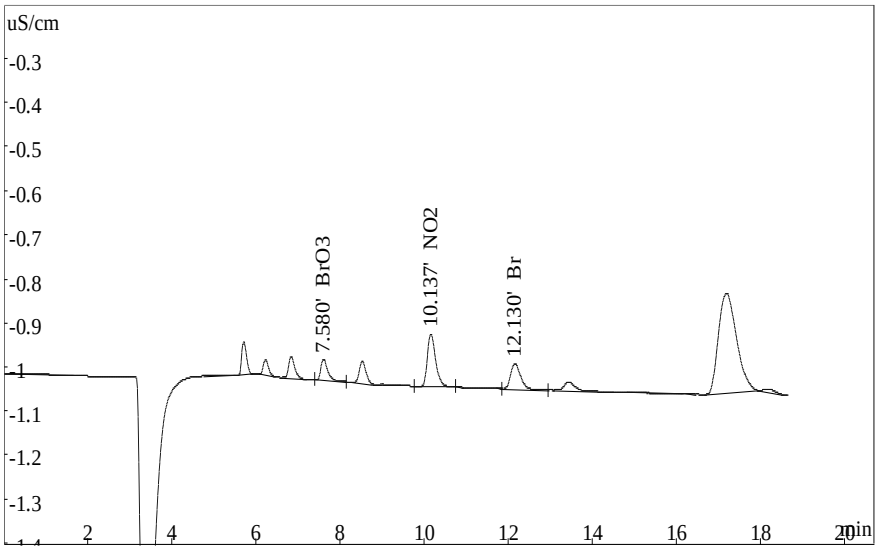
Chrotrogram of nitrite and nitrate ion in pumpkin powder

1.10 Determination of bromate, nitrite and bromide in purified water

Pure water is the natural water through multiple processes of treatment, purification and purification of water. The limit of bromate is 0.01 mg / L in GB 5749-2006 hygienic standard for drinking water, and the limit of nitrite in bottled (barrel) purified water is 0.002 in GB 17324-2003 Ion chromatography can accurately and quickly detect low content bromate and nitrite in purified water, which provides a powerful means for water quality monitoring.

Analysis Conditions:

- Analytical Column：SH-G-1+SH-AP-1
- Mobile Phase：15 mM KOH（EG）
- Flow Rate：0.7 mL/min
- Column Temperature：35℃
- Injection Volume：200 µL
- Suppressor：SHY-A-6
- Pretreatment：The sample was injected into 0.22 µ m disposable needle filter for analysis.



Chromatogram of bromate, nitrite and bromine at 10 µ g / L

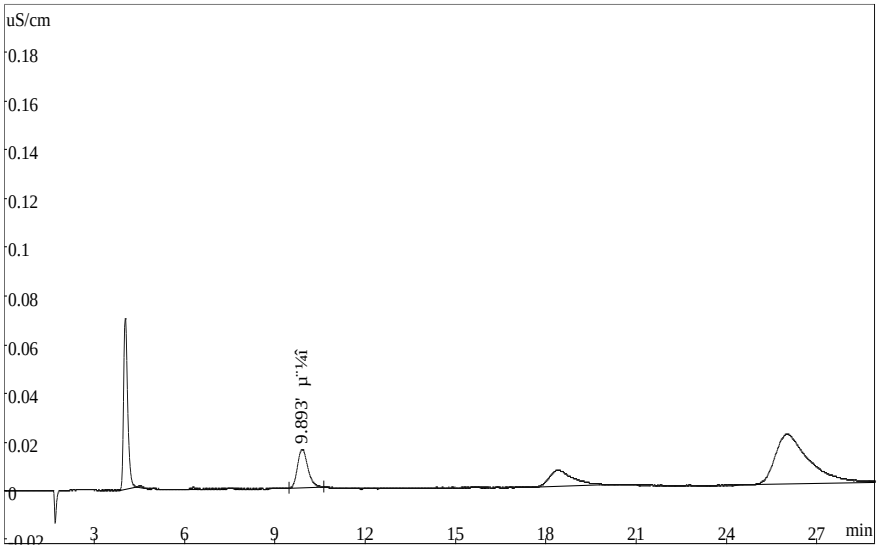
1.11 Determination of choline

Choline is a component of all biofilms. Choline is obtained from food and PC (via continuous methylation of phosphatidylethanolamine (PE)). Choline plays an important role in promoting brain development and memory ability, ensuring information transmission and promoting fat metabolism. Infant formula milk powder and other foods will be added choline, appropriate intake is beneficial to the body.

Analysis Conditions:

- Analytical Column：SH-G-1+SH-CC-4
- Mobile Phase：4.0 mM MSA（EG）
- Flow Rate：1.0 mL/min
- Column Temperature：35℃
- Injection Volume：10 μL
- Suppressor：SHY-C-3
- Pretreatment：Weigh 0.06 g (accurate to 0.1 mg) of sample into a 100 ml plastic volumetric flask, dissolve it with high-purity water, mix it up to the scale, dilute it 100 times, and then inject the sample through 0.22 μ m disposable needle filter

for determination.



Chromatogram of choline in the sample

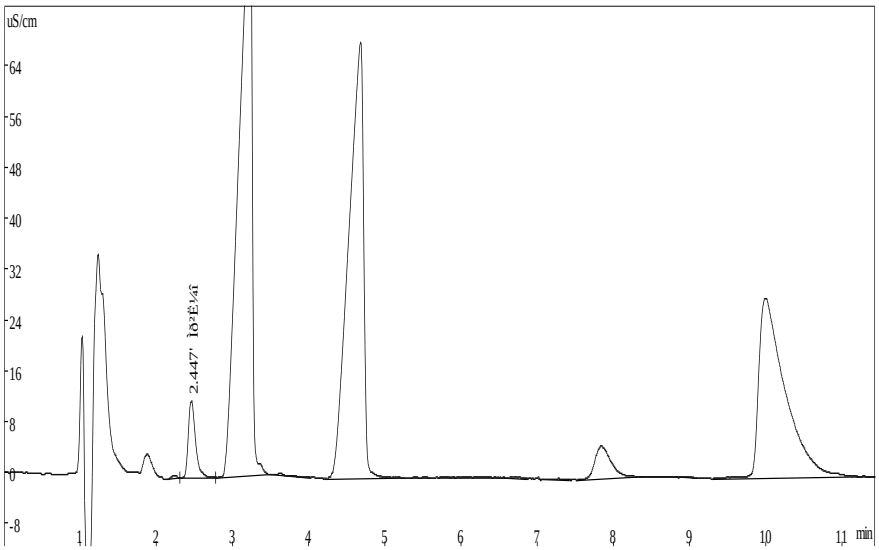
1.12 Determination of betaine

Betaine is a kind of alkaloid with strong hygroscopicity. Its molecular structure and application effect are similar to that of natural betaine. Betaine belongs to the

chemical synthetic natural substance equivalent. As a feed additive, betaine can provide methyl donor function, regulate osmotic pressure in vivo, alleviate stress, promote fat metabolism and protein synthesis, improve lean meat rate, and enhance the efficacy of anticoccidial drugs. Betaine can stimulate the sense of smell and taste of animals, and is used as food attractant in aquatic animal feed.

Analysis Conditions:

- Analytical Column：SH-G-1+SH-YS-50
- Mobile Phase：4.0 mM MSA（EG）
- Flow Rate：1.0 mL/min
- Column Temperature：40℃
- Injection Volume：25 μL
- detection method：Direct Conductometry
- Pretreatment: after 10 times dilution, the sample is injected into 0.22 μ m disposable needle filter for determination.



Chromatogram of betaine in sample