RFIC

Determination of chloride in multivitamin-mineral supplements using ion chromatography

Authors

Hua Yang and Jeffrey Rohrer
Thermo Fisher Scientific, Sunnyvale, CA

Keywords

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Goal

To develop a method for the determination of chloride in multivitamins using an ion chromatography (IC) system with suppressed conductivity detection

Introduction

Chlorine is an essential element for humans. Chloride is one of the most important electrolytes in the blood[1], and an appropriate level is critical for human health. Most of the chloride in our body comes from the salt (sodium chloride) we eat, and many dietary supplements also contain chloride. Knowing the chloride content of foods is important, and determination of chloride in food or dietary supplements has been included in the National Institute of Standards and Technology (NIST) Dietary Supplement Laboratory Quality Assurance Program (DSQAP).[2]

Chloride is commonly determined by IC. This application brief shows a 15-min IC method for the determination of chloride in multivitamin-mineral supplement samples. This method uses alkaline digestion to prepare the sample, and a 2 mm version of a Thermo Scientific™ Dionex™ IonPac™ AS20 column with electrolytically generated potassium hydroxide (KOH) eluent. Using an alkaline digestion sample preparation method and a dual channel IC system, the nutrient iodine (as iodide) in the same sample can be determined by electrochemical detection (ED) simultaneously. The determination of iodine in these samples was published in a recent application note.[3]

Experimental

Equipment and consumables

- Thermo Scientific™ Dionex™ ICS-6000 Dual Channel HPIC™ System with RFIC-EG module, Conductivity and Electrochemical detectors*
- Thermo Scientific™ Dionex™ AS-AP Autosampler with 250 µL syringe and tray temperature control
- Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, version 7.2.10

*For chloride determination, equivalent results can be achieved using a single channel Thermo Scientific™ Dionex™ ICS-6000 system, Thermo Scientific™ Dionex™ ICS-5000+ system, or Thermo Scientific™ Dionex™ Integrion™ HPIC™ system with conductivity detection.

Consumables

- Thermo Scientific™ Dionex™ EGC 500 KOH Potassium Hydroxide Eluent Generator Cartridge (P/N 075778)
- Thermo Scientific™ Dionex™ CR-ATC 600 Continuously Regenerated Anion Trap Column (P/N 088662)
- Thermo Scientific™ Dionex™ IonPac™ AS20 Analytical Column, 2 x 250 mm (P/N 063065)
- Thermo Scientific™ Dionex™ IonPac™ AG20 Guard Column, 2 x 50 mm (P/N 063066)
- Dionex AS-AP Autosampler Vials, 10 mL (P/N 074228)
- Fisherbrand™ Polypropylene Centrifuge Tubes, 50 mL (Cat. No. 05-539-13)
- Thermo Scientific™ Nalgene™ Syringe Filter 0.2 µm PES (Cat. No. 725-2520)

Reagent and standards

- Degassed deionized (DI) water, 18 MΩ·cm resistance or better
- Sodium chloride (>99.9%), A.C.S. reagent grade or better, for preparing chloride standards
- Sodium hydroxide solution (50% w/w Certified), Fisher Chemical™ (Fisher Scientific P/N SS254-500)

Samples

Four brands of multivitamins were purchased from local stores.

Table 1. Multivitamin samples

Multivitamin	Information
Sample 1 (MV1)	Brand 1, Multivitamin for men
Sample 2 (MV2)	Brand 2, Multivitamin for women
Sample 3 (MV3)	Brand 3, Organic multivitamin
Sample 4 (MV4)	Brand 4, Multivitamin

Experimental setup

Figure 1 shows a schematic diagram of a Reagent-Free™ IC (RFIC) system for the determination of chloride here. The IC system is also used for the determination of iodide (AN000069).[3] An autosampler with a diverter valve is used to serve both channels. The multivitamin samples are analyzed by CD and ED simultaneously. For the determination of chloride, only one pump of the DP or a single pump is used and the diverter valve is not needed for the Dionex AS-AP autosampler.

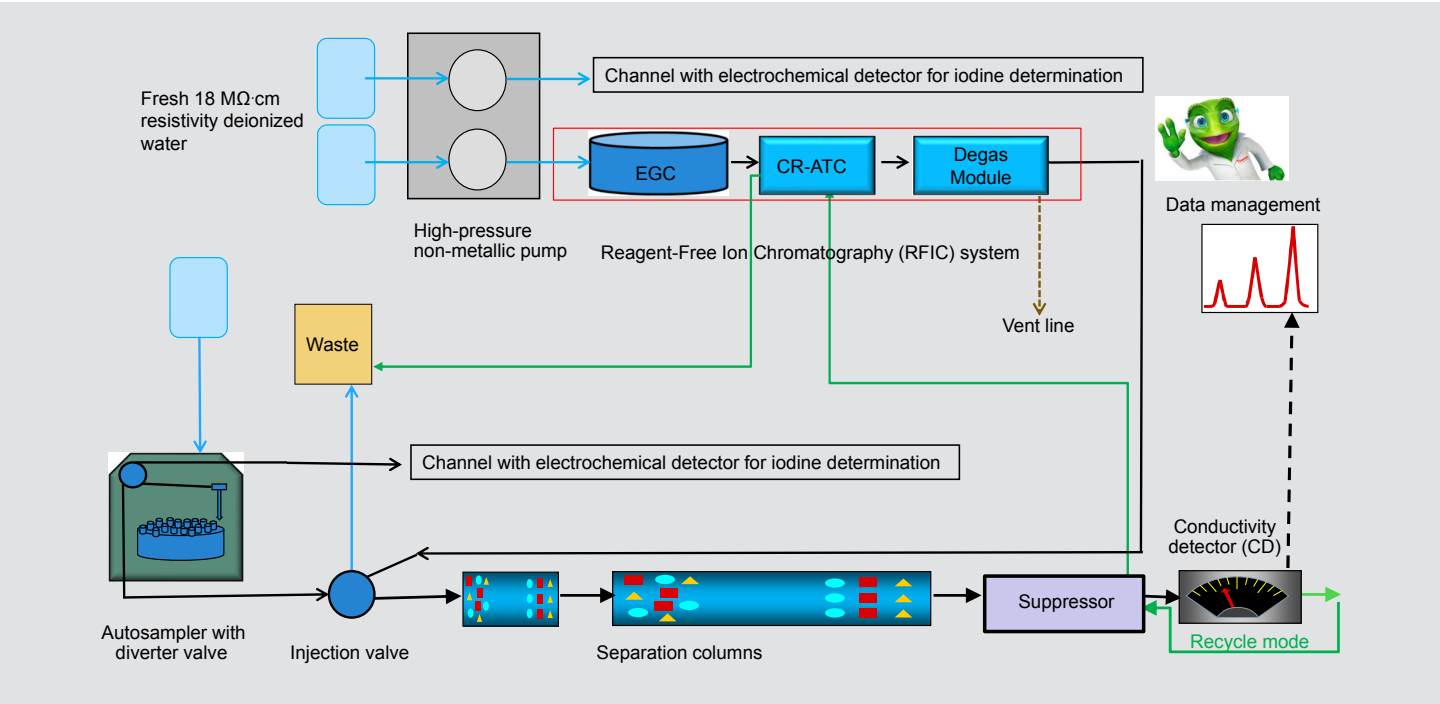

Figure 1. Schematic diagram of an IC system for the determination of chloride

Preparation of solutions and reagents

Calibration standards

Chloride stock standard (2,000 mg/L) solution is prepared by dissolving 330 mg of dry sodium chloride salt in 100 mL of DI water. The stock standard is stable for at least six months when stored at 4 ˚C. Diluted calibration standard solutions are prepared from either the 2,000 mg/L stock standard or the diluted 10 mg/L calibration standard, and DI water.

Sample preparation

Extraction solution (0.02% (w/w) sodium hydroxide): Mix 0.4 g of 50% NaOH with 999.6 g DI water in a plastic bottle.

Sample powder: Grind >30 tablets and mix the resulting powder thoroughly. Store at room temperature if analyzed within 2 days. Otherwise, store at 4 ˚C.

Prepare sample:

1. Weigh about 0.500 g of multivitamin sample into a 50 mL conical centrifuge tube, record exact weight.
2. Add 20 mL (g) of extraction solution into the tube, shake, and mix well.
3. Place the tube in an ultrasonic bath and sonicate for ≥2 h.
4. Centrifuge at 6,000-7,000 rpm for 30 min.
5. Filter the sample solution through a 0.2 µm PES syringe filter, discarding the first couple drops of the effluent.
6. Dilute the sample solution 1 to 20 with the extraction solution before IC analysis.

Results and discussion

Separation

The Dionex IonPac AS20 column is a hydroxide-selective high capacity anion-exchange column developed to determine anions that are strongly retained on other anion-exchange columns. With this column, highly retained anions, such as iodide, can be determined with weaker eluents and in less time compared to other anion-exchange columns[4]. The method to determine chloride in multivitamin-mineral supplements includes two steps: 8 min of 10 mM KOH for separation and 5 min of 65 mM KOH for column cleaning, which ensures strongly retained anions elute and do not consume column capacity. Figure 2 shows the separation of chloride in the four multivitamin samples. Chloride elutes at 5.4 min and is well-resolved from other anions in the samples.

Linearity and method detection limit

The method linearity was determined by triplicate injections of seven levels of calibration standards (Table 2). The results are shown in Table 3 and Figure 3. The study shows that peak area response is linear over the concentration range from 0.2 to 500 mg/L with r^2=1.000.

Table 2. Calibration standards (mg/L)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Chloride (mg/L)	0.2	2	10	50	100	200	500

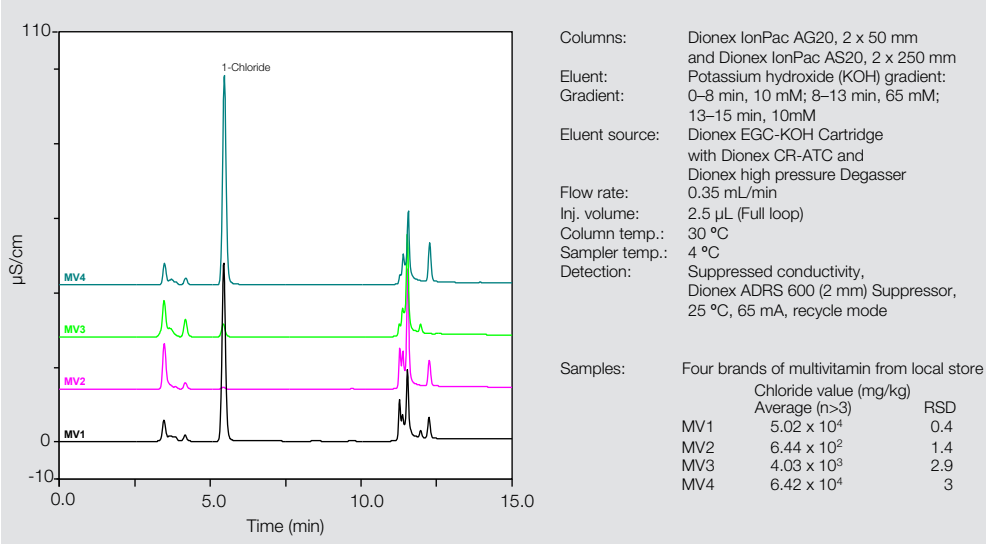

Figure 2. Chromatograms of multivitamin samples

Table 3. Linearity for chloride determination

Analyte	Injection volume (µL)	Range (mg/L)	Coefficient of determination* (r^2)
Chloride	2.5	0.2-500	1.0000

* Calibration type is linear and forced through the origin.

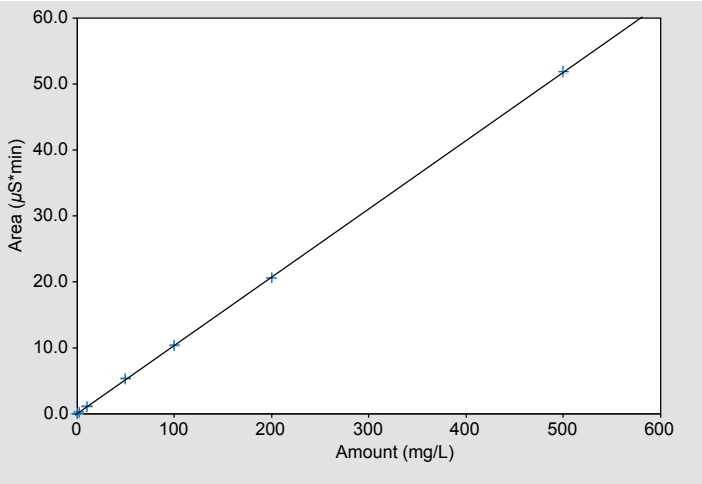

Figure 3. Calibration plot for chloride

Determination of chloride in multivitamin and precision

The method precision was evaluated by measuring chloride in four different multivitamin samples and expressed as the relative standard deviation (RSD) of the results (Table 4). Interday precision was evaluated by comparing the results over 3 days with new sample preparations each day. The method is precise with interday precision less than 3.0%. There were no label values for chlorine to judge accuracy.

Table 4. Chloride in multivitamin samples

Multivitamin	Measured chloride (mg/kg)	RSD
MV1	5.02×10^4	0.4
MV2	6.44×10^2	1.4
MV3	4.03×10^3	2.9
MV4	6.42×10^4	3.0

Conclusion

This application brief demonstrates a 15 min IC method for the determination of chloride in multivitamin-mineral supplements. The method uses a 2 mm Dionex IonPac AG20/AS20 column set with electrolytically generated potassium hydroxide (KOH) eluent. Using this method, chloride is well resolved from other anions found in the multivitamin samples. The method has a linear calibration over the concentration range from 0.2 to 500 mg/L with r^2=1.000 and the method is precise (RSD ≤ 3.0%). Four multivitamin samples were tested, and the data show this IC-CD method can be used for the determination of chloride in multivitamins.

References

1. Terry J. The major electrolytes: sodium, potassium, and chloride. J Intraven Nurs. 17:240–247.
2. Dietary Supplement Laboratory Quality Assurance Program https://www.nist.gov/programs-projects/dietary-supplement-laboratory-quality-assurance-program (Accessed April 8, 2021.)
3. Thermo Scientific Application Note 000069, Determination of iodide in multivitamin-mineral supplements using ion chromatography, Sunnyvale, CA, USA, 2021 https://appslab.thermofisher.com/App/4575/determination-iodide

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