

## Application News

### Ion Chromatograph

# Simultaneous Analyses of Anions and Cations in Nutrient Solution Used for Hydroponic Cultivation and in Its Dilution Water (Source Water) Using the Nexera™ IC Ion Chromatograph

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### User Benefits

- ◆ Simultaneous analyses of anions and cations in nutrient solution used for hydroponic cultivation and in its dilution water (hereinafter referred to as source water) can be performed.
- ◆ Different injection volumes can be set for anion and cation analyses, respectively.
- ◆ Consolidated data management can be done since analytical results for anions and cations are saved in one data file.

### ■ Introduction

In order to maintain stable plant growth, hydroponic cultivation is generally controlled by pH and electrical conductivity (EC) of nutrient solution as indicators. While pH indicates acidity or alkalinity of nutrient solution and EC indicates the total amount of dissolved ionic substances, it is difficult to verify whether appropriate balances of individual nutrient levels are accomplished or not based on pH and EC alone. It is important to analyze nutrient solution regularly to determine the concentration of each nutrient since unbalanced nutrient levels often provide poor growth and nutrient deficiencies. Ion chromatography is an effective method for individual determination of anions and cations in nutrient solution.

Furthermore, it is important to verify the quality of the source water since the ionic composition of the source water used for nutrient solution also influences its initial composition and management methods.

In this article, the analyses of five types of anions and five types of cations in nutrient solution used for hydroponic cultivation and in its source water were presented, which were performed using Nexera IC dual analysis system (hereinafter referred to as the 'dual analysis system').

### ■ Analysis of Mixed Standard Solution

Chromatograms of a mixed standard solution of five anions (chloride ion, nitrite ion, nitrate ion, phosphate ion and sulphate ion) and a mixed standard solution of five cations (sodium ion, ammonium ion, potassium ion, magnesium ion and calcium ion) are shown in Fig. 1. The analytical conditions are shown in Table 1. Five-level calibration curves were created using mixed standard solutions. Table 2 shows the concentrations of anions and cations in mixed standard solutions and coefficients of determination ( $r^2$ ) of calibration curves, which showed 0.999 or more for all ions, resulting in good linearities.

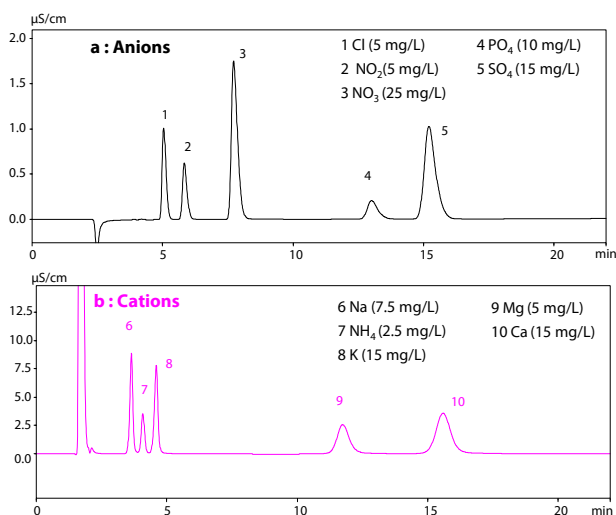


Fig. 1 Chromatograms of mixed standard solutions  
(a: anions b: cations)

Table 1 Analytical conditions

|                  |                                                                     |
|------------------|---------------------------------------------------------------------|
| System           | Nexera IC                                                           |
| Anion Column     | Shim-pack IC-SA2 <sup>*1</sup><br>(250 mm × 4.0 mm I.D., 9 μm)      |
| Guard Column     | Shim-pack IC-SA2(G) <sup>*2</sup><br>(10 mm × 4.6 mm I.D., 9 μm)    |
| Suppressor       | ICDS-Ai                                                             |
| Mobile phase     | 1.8 mmol/L Sodium Carbonate<br>1.7 mmol/L Sodium Hydrogen Carbonate |
| Flow rate        | 1.0 mL/min                                                          |
| Column temp.     | 30 °C                                                               |
| Injection volume | 10 μL (Sample consumption volume : 28 μL)                           |
| Injection type   | Partial loop (50 μL loop)                                           |
| Cation Column    | Shim-pack IC-C4 <sup>*3</sup><br>(150 mm × 4.6 mm I.D., 7 μm)       |
| Guard Column     | Shim-pack IC-C4(G) <sup>*4</sup><br>(8 mm × 3 mm I.D., 7 μm)        |
| Mobile phase     | 2.5 mmol/L Methanesulfonic Acid                                     |
| Flow rate        | 1.0 mL/min                                                          |
| Column temp.     | 40 °C                                                               |
| Injection volume | 25 μL (Sample consumption volume : 59 μL)                           |
| Injection Type   | Partial loop (50 μL loop)                                           |
| Vial             | Shimadzu PP vial for 1.5 mL <sup>*5</sup>                           |
| Detection        | Conductivity Detector                                               |

\*1 P/N : 228-38983-91

\*2 P/N : 228-38983-92

\*3 P/N : 228-41616-91

\*4 P/N : 228-59900-91

\*5 P/N : GLC-IVS-100

Table 2 Concentrations of mixed standard solutions and coefficients of determination of corresponding calibration curves

| Target ion | Concentration(mg/L) | $r^2$            |
|------------|---------------------|------------------|
| Anion      | Cl                  | 0.5/1/2.5/5/10   |
|            | NO <sub>2</sub>     | 0.5/1/2.5/5/10   |
|            | NO <sub>3</sub>     | 2.5/5/12.5/25/50 |
|            | PO <sub>4</sub>     | 1/2/5/10/20      |
|            | SO <sub>4</sub>     | 1.5/3/7.5/15/30  |
| Cation     | Na                  | 0.3/1.5/3/7.5/15 |
|            | NH <sub>4</sub>     | 0.1/0.5/1/2.5/5  |
|            | K                   | 0.6/3/6/15/30    |
|            | Mg                  | 0.2/1/2/5/10     |
|            | Ca                  | 0.6/3/6/15/30    |

### ■ Partial Loop Injection and Full Loop Injection

The dual analysis system employs the loop injection method. Fig. 2 shows the flow path diagram of the system. In the loop injection method, the partial loop mode and the full loop mode can be selected. The partial loop mode requires filling a portion of the sample loop with the sample, then switching the high-pressure valve to introduce the sample into the column. On the other hand, the full loop mode requires completely replacing the sample loop with the sample, then switching the high-pressure valve to introduce a volume of sample equal to that of the sample flow path involving the sample loop into the column. As the injection volume can be changed in the partial loop mode, the optimal injection volume can be set according to the concentrations of ions.

In this article, the partial loop mode was employed to inject adjusted volumes to suit the concentration ranges of anions and cations.

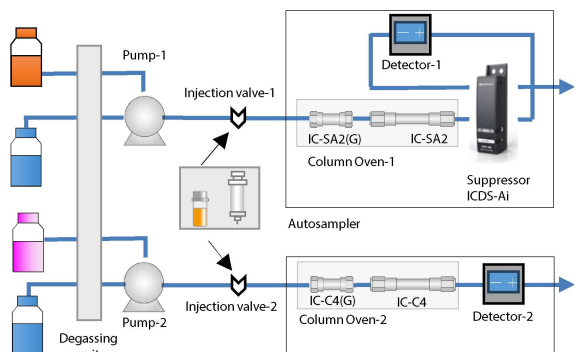


Fig. 2 Flow path diagram of dual analysis system of ion chromatograph

## Real Sample Analysis

As real samples, tap water was used as the source water, and a nutrient solution was prepared by diluting a commercially available liquid fertilizer 500-fold with tap water, as specified by the product instructions.

The tap water was filtered through a 0.2 µm syringe filter (for ion chromatography: P/N 227-33306-02) before analysis. The nutrient solution was diluted ten-fold with ultrapure water, filtered, and then analyzed. Fig. 3 shows the chromatograms of the tap water, and Fig. 4 shows the chromatograms of the nutrient solution. Table 3 shows the average concentrations obtained from six consecutive analyses of each sample, and Table 4 shows the repeatability of each peak area. By setting different injection volumes for anions and cations, 10 µL and 25 µL respectively, both ion groups with different concentration ranges were analyzed with favorable peak shapes.

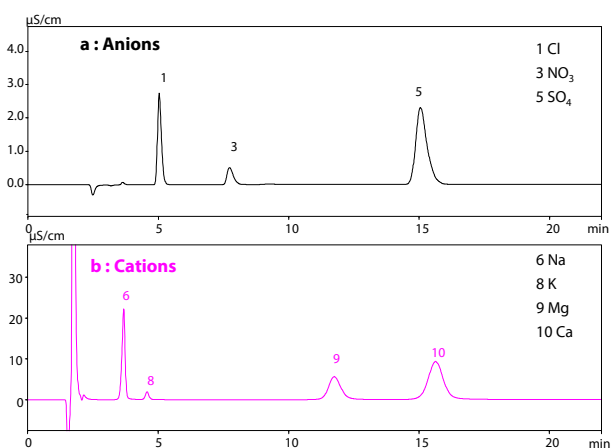


Fig. 3 Chromatograms of tap water (a: anions b: cations)

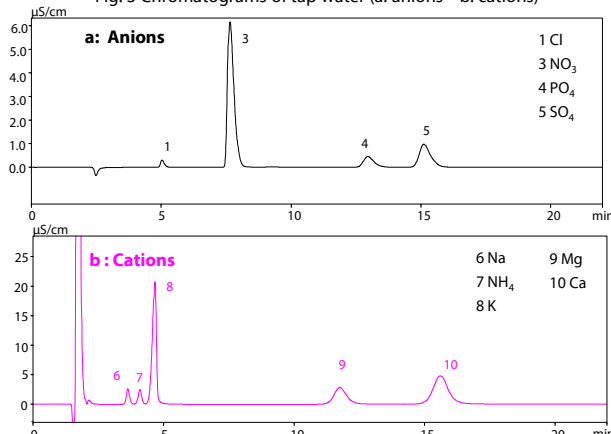


Fig. 4 Chromatograms of ten-fold diluted nutrient solution (a: anions b: cations)

Since EC indicates the total amount of dissolved ions in nutrient solution, it is not possible to individually determine the increase or decrease of components with largely different concentration ranges, such as nitrate ion and potassium ion. In this analysis, it was confirmed that nitrate ion and potassium ion were the major components in the nutrient solution, and that the tap water used as the source water also contained calcium ion at 15.4 mg/L, sulphate ion at 16.2 mg/L, and other components, providing information on each component that cannot be obtained by EC-based management alone. For components contained in both the source water and the fertilizer, the analysis of the source water together with the nutrient solution is considered useful, since the quality of the source water influences the initial composition of the nutrient solution.

Table 3 Determination results (Average concentrations)  
(Unit: mg/L n=6, Equivalent to non-diluted nutrient solution)

| Target ion | Tap water       | Nutrient solution <sup>*1</sup> |
|------------|-----------------|---------------------------------|
| Anion      | Cl              | 6.5                             |
|            | NO <sub>2</sub> | N.D. <sup>*2</sup>              |
|            | NO <sub>3</sub> | 437.7                           |
|            | PO <sub>4</sub> | 105.6                           |
|            | SO <sub>4</sub> | 70.3                            |
| Cation     | Na              | 9.1                             |
|            | NH <sub>4</sub> | 7.4                             |
|            | K               | 172.2                           |
|            | Mg              | 21.6                            |
|            | Ca              | 79.3                            |

\*1 Conversion formula: Actual value x dilution factor (10)

\*2 N.D.: not detected

Table 4 Peak area repeatabilities (n=6, %RSD)

| Target ion | Tap water       | Nutrient solution |
|------------|-----------------|-------------------|
| Anion      | Cl              | 0.46              |
|            | NO <sub>2</sub> | - <sup>*1</sup>   |
|            | NO <sub>3</sub> | 0.19              |
|            | PO <sub>4</sub> | 0.20              |
|            | SO <sub>4</sub> | 0.18              |
| Cation     | Na              | 0.26              |
|            | NH <sub>4</sub> | 0.38              |
|            | K               | 0.18              |
|            | Mg              | 0.26              |
|            | Ca              | 0.26              |

\*1 -: Not calculated because the result was N.D.

## Conclusion

Simultaneous analyses of anions and cations in nutrient solution used for hydroponic cultivation and in the source water using the dual analysis system have been introduced. As the system allows individual injection volume settings for the anion and cation flow paths, simultaneous analyses of anions and cations can be performed considering the concentration ranges of the respective anion and cation groups.

Efficient monitoring of individual ions in nutrient solution and in its source water helps to review the fertilization design and to determine the timing of nutrient replenishment. Although nitrite ion was not detected in any of the samples, it may accumulate in nutrient solution and cause poor plant growth. In addition, when environmental water is used as the source water, nitrite ion may be present at concentrations that affect plant growth. Therefore, nitrite ion can also be used as a management parameter.

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