

# Determination of Total Inorganic Arsenic in Fruit Juice Using High-Pressure Capillary Ion Chromatography

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## Key Words

Conductivity Detection, Food and Beverage, High Resolution, Total Inorganic Arsenic, 4  $\mu\text{m}$  Column

## Goal

Demonstrate the determinations of total inorganic arsenic in fruit juice using high-pressure capillary Ion Chromatography (IC).

## Introduction

Growing interest around arsenic (As) determinations in fruit juices has been triggered by media claims of total arsenic concentrations above acceptable limits in apple juice products.<sup>1</sup> Although the FDA has been testing and monitoring fruit juices for arsenic content for more than 20 years and has found that total inorganic arsenic levels in juice are typically low, more recently there has been heightened scrutiny of arsenic in apple juice.<sup>2,3</sup> The U.S. EPA has set the total arsenic standard for drinking water at 0.010 parts per million (ppm).<sup>4</sup> However, total arsenic determinations can be misleading because inorganic arsenic compounds (arsenate As(V) and arsenite As(III)) are highly toxic, whereas organic arsenic compounds have much lower toxicity.<sup>5,6</sup> The established analytical method for the various arsenic species is separation by IC followed by detection using Inductively Coupled Plasma Mass Spectrometry (ICP-MS).<sup>7,8</sup> IC with suppressed conductivity detection (CD) is a well established, complimentary method used by multiple industries for ionic determinations.<sup>9–11</sup> This method has been demonstrated to be an excellent way to characterize fruit juices and wine.<sup>12,13</sup> The recently introduced high-pressure capable capillary IC systems combined with 4  $\mu\text{m}$  particle ion-exchange columns have reduced eluent consumption and further improved separation efficiency. This study presents the detection and quantification of total inorganic arsenic (Arsenate As(V) and Arsenite As(III)) in fruit juices separated on a high-resolution 4  $\mu\text{m}$  particle anion-exchange column using a high-pressure capillary IC system.



## Equipment

Thermo Scientific™ Dionex™ ICS-5000+ HPIC™ high-pressure capillary IC system:

- Thermo Scientific Dionex ICS-5000+ DC Detector/Chromatography module with high-pressure degas cartridge
- Thermo Scientific™ Dionex™ IC Cube™
- Thermo Scientific Dionex ICS-5000+ DP Dual Pump module with high-pressure capillary pumps
- Thermo Scientific Dionex ICS-5000+ EG Eluent Generator module
- Thermo Scientific Dionex AS-AP Autosampler
- Thermo Scientific™ Dionex™ Chromeleon™ 7.2 Chromatography Data system (CDS) software

## Reagents and Standards

- Stock standards: 1000 mg/L of individual anion stock standards were prepared by dissolving acids or salts in 18 M $\Omega$ -cm resistivity deionized water. Reagent needed to prepare 1 L of anion stock are listed in Table 1
- Mixed working standard (mixed SD): A working standard solution was prepared by mixing the stock standards to the concentrations indicated in Table 1 with deionized water
- Arsenate calibration standards: The arsenate calibration standards were prepared by diluting the arsenate stock solution with deionized water. 0.2, 0.5, 1.0, 4.0, 5.0, 10, 25, 50, and 100 mg/L arsenate standards were used
- All ACS grade reagents were purchased from Fisher Scientific

The amounts of compounds used to prepare 1 L of 1000 mg/L anion stock standards and the anion concentrations in mixed working standard are described below (Table 1).

## Sample Preparation

Commercial juice samples were filtered with a syringe filter (0.20  $\mu$ m), and then diluted 50-fold with deionized water. Fifty-fold diluted juice samples were spiked with 0.19 and 0.50 mg/L arsenate.

Note: It is important to use 18 M $\Omega$ -cm resistivity, deionized water for standard, eluent, and autosampler flush solutions to avoid system contamination, decreased sensitivity, and poor calibration. Degassing the deionized water by vacuum filtration prior to use is a good practice.

Table 1. Standard preparation table.

Anion	Reagent	Weight* (g)	Mixed SD (mg/L)	Anion	Reagent	Weight* (g)	Mixed SD (mg/L)
Quinate	Quinic acid	1.000	9.3	Succinate	Sodium succinate	1.396	10.1
Fluoride	Sodium fluoride	2.210	2.3	Malate	L-Malic acid	1.000	–
Lactate	Lithium lactate	1.067	16.7	Malonate	Malonic acid	1.000	9.6
Acetate	Sodium acetate, trihydrate	2.305	11.4	Tartrate	Sodium tartrate	1.311	–
Glycolate	Glycolic acid	1.000	–	Maleate	Maleic acid	1.000	7.9
Propionate	Sodium propionate	1.315	8.6	Sulfate	Sodium sulfate	1.479	10.0
Formate	Sodium formate	1.511	10.4	Oxalate	Sodium oxalate	1.522	8.9
Butyrate	Sodium butyrate	1.250	9.4	Fumarate	Fumaric acid	1.000	9.0
Pyruvate	Pyruvic acid	1.000	10.8	Phosphate	Potassium phosphate, monobasic	1.433	9.3
Valerate	Valeric acid	1.000	–	Citrate	Citric acid	1.000	25.4
Galacturonate	D-Galacturonic acid, monohydrate	1.000	13.2	Isocitrate	Isocitric acid, trisodium dihydrate	1.306	9.5
Bromate	Sodium bromate	1.179	7.8	<i>cis</i> -Aconitate	<i>cis</i> -Aconitic acid	1.000	11.4
Chloride	Sodium chloride	1.648	4.0	<i>trans</i> -Aconitate	<i>trans</i> -Aconitic acid	1.000	11.8
Nitrite	Sodium nitrite	1.000	20.1	Arsenate	Sodium arsenate, dibasic heptahydrate	2.246	9.4
Bromide	Sodium bromide	1.288	10.1	Arsenite	Sodium meta-arsenite	1.215	–
Nitrate	Sodium nitrate	1.371	11.5				
Glutarate	Glutaric acid	1.000	13.1				

## Conditions

### Ion Chromatography

Instrument:	Dionex ICS-5000+ HPIC system
Column:	Thermo Scientific™ Dionex™ IonPac™ AS11-HC-4 $\mu$ m column (0.4 $\times$ 250 mm)
Column Temp.:	30 °C
IC Cube Temp.:	15 °C
Eluent Source:	Thermo Scientific Dionex EGC–KOH Cartridge (Capillary)
Gradient:	1.5 mM KOH (–10–0 min); 1.5–2 mM KOH (0–2 min); 2–8 mM (2–13 min); 8–28 mM (13–25 min); 28–35 mM (25–33 min); 35–65 mM (33–34 min); 65 mM (34–38 min)
Flow Rate:	0.015 mL/min
Inj. Volume:	0.40 $\mu$ L
Detection:	Suppressed conductivity, AutoSuppression™ Thermo Scientific™ Dionex™ ACES™ 300 Anion Capillary Electrolytic Suppressor, recycle mode, 13 mA
System Backpressure:	~3000 psi

Part numbers of the consumables used in this document are listed in Table 2.

\* Reagent needed to prepare 1 L of 1000 mg/L anion stock.

Table 2. Consumables list.

Product Name	Description	Part Number
Dionex EGC-KOH cartridge (Capillary)	Eluent Generator cartridge	072076
Thermo Scientific Dionex CR-ATC Continuously Regenerating Anion Trap Column (Capillary)	Electrolytic capillary trap column	072078
Dionex IonPac AS11-HC-4 $\mu$ m column	Separation column	078031
Thermo Scientific Dionex CRD 180 Carbonate Removal Device (Capillary)	Carbonate removal device cartridge	079960
Dionex ACES 300 Anion Capillary Electrolytic Suppressor	Suppressor cartridge	072052
Dionex HP fittings (blue)	Bolts/Ferrules	074449/ 074373
EG Degas HP cartridge	High-pressure degas cartridge, up to 5000 psi	AAA-074459
Dionex AS-AP Autosampler vials	Package of 100, 10 mL polystyrene vials, caps, blue septa	074228
Dionex IonPac ATC-500 column	Anion trap column	079018

### Instrument Setup and Installation

To achieve the best chromatography with capillary IC, it is important to minimize void volumes in each connection by using precision cut tubing, high pressure connectors and fittings (blue color), and seating the ferrule >2 mm above the end of the tubing. Extra care should be used to prevent air in all consumables or tubing by observing steady liquid flow before installing the next device in line. A thorough discussion can be found in *Thermo Scientific Technical Note (TN) 113: Practical Guidance for Capillary IC*.<sup>14</sup>

Figure 1 shows the flow diagram of this application. EG Degas HP, Dionex Carbonate Removal Device, and Dionex ACES suppressor cartridges and both columns are installed in the Dionex IC Cube (Figure 2). Prior to use, the Dionex ACES suppressor and Dionex CR-ATC trap column should be hydrated, and the Dionex EGC cartridge and Dionex IonPac columns should be conditioned. The instructions can be found in section 3.18 of the Dionex ICS-5000<sup>+</sup> HPIC system installation manual.<sup>15</sup> Detailed instructions are also described in *Thermo Scientific TN 131: Configuring High-Pressure Capillary IC on the Modular IC System*, the product manuals, and the instrument installation and operator's manuals.<sup>14-19</sup>

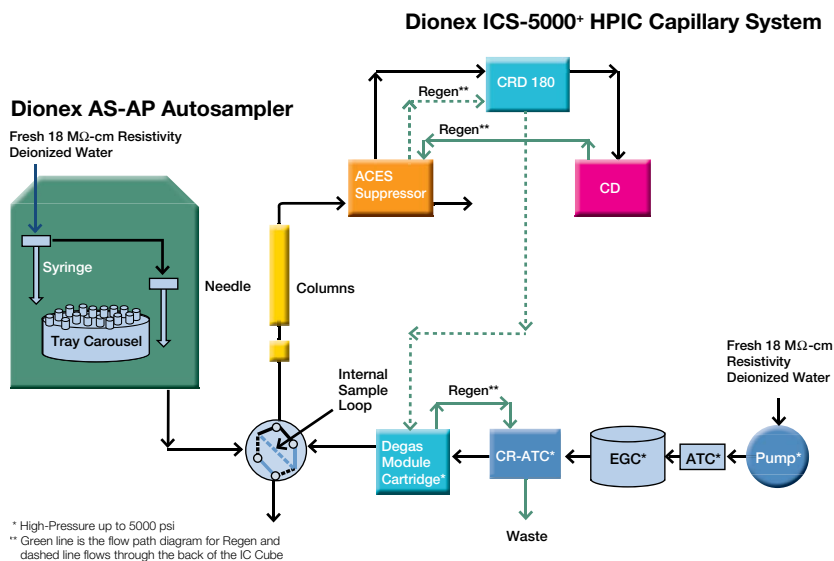


Figure 1. Flow diagram.

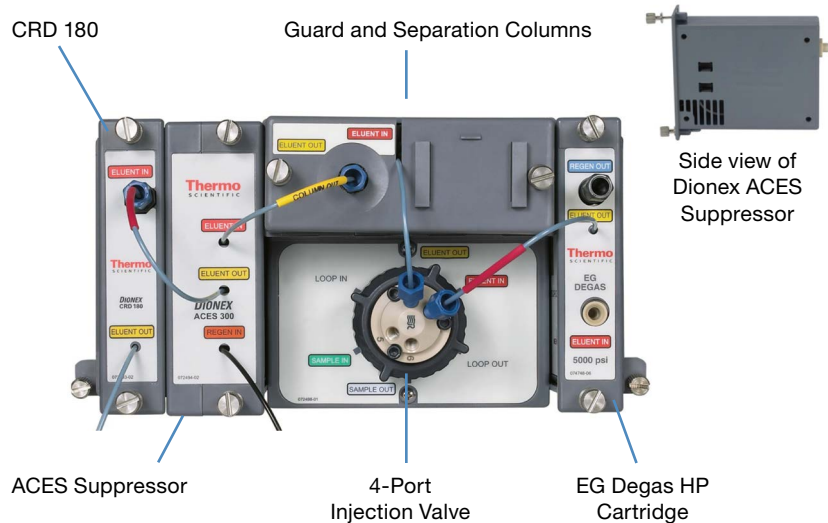


Figure 2. Dionex IC Cube.

## Results and Discussion

The Dionex IonPac AS11-HC columns are well known for their ability to separate a large variety of anions, including organic and inorganic acids in fruit juices and wine.<sup>12, 13</sup> The newly introduced 4  $\mu\text{m}$  resin particle technology has further increased chromatographic efficiency and improved resolution with higher operating pressures. A Dionex ICS-5000+ HPIC system, capable of handling pressures up to 5000 psi, and a Dionex IonPac AS11-HC-4 $\mu\text{m}$  capillary column were selected for this study.

Ion chromatography with suppressed conductivity detection has been demonstrated to be an excellent way to characterize fruit juices and wine. With minimum modification, such as adding shallow gradients at the beginning to increase resolution of early eluting peaks and between 28 to 35 minutes to achieve a baseline resolution of arsenate and citrate, a less than 40 minute method was developed for arsenic analysis.

The chromatogram in Figure 3 is a mixed working standard for fruit juice analysis. This shows the separation of 27 organic and inorganic acids in a single injection. However, some critical pairs, such as glycolate/acetate, valerate/galacturonate, succinate/malate, and malonate/tartrate, cannot be separated without adding an organic modifier. Coelutions were proved by injection of individual acid standard and their mixture. Acetate, galacturonate, succinate and tartrate were selected for inclusion in the standard mix due to their likelihood to be in juices. With this method, arsenate eluted at 32.9 minutes (Peak 23) and was well resolved from citrate (Peak 24) indicating that the parameters used are appropriate for arsenate analysis in fruit juices.

Column: Dionex IonPac AS11-HC-4 $\mu\text{m}$  column, 0.4  $\times$  250 mm  
 Instrument: Dionex ICS-5000+ HPIC system  
 Eluent Source: Dionex EGC-KOH cartridge (Capillary)  
 Gradient (KOH): 1.5–2 mM (-10–2 min), 2–8 mM (2–13 min), 8–28 mM (13–25 min), 28–35 mM (25–33 min), 35–65 mM (33–34 min), 65 mM (34–38 min)  
 Flow Rate: 0.015 mL/min  
 Inj. Volume: 0.40  $\mu\text{L}$   
 Column Temp.: 30  $^{\circ}\text{C}$   
 IC Cube Temp.: 15  $^{\circ}\text{C}$   
 Detection: Suppressed conductivity, Dionex ACES 300 Suppressor, AutoSuppression, recycle mode

Peak	mg/L	Peak	mg/L
1. Quinate	9.3	15. Glutarate	13.1
2. Fluoride	2.3	16. Malate/Succinate	10.1
3. Lactate	16.7	17. Malonate/Tartrate	9.6
4. Acetate	11.4	18. Maleate	7.9
5. Propionate	8.6	19. Sulfate	10.0
6. Formate	10.4	20. Oxalate	8.9
7. Butyrate	9.4	21. Fumarate	9.0
8. Pyruvate	10.8	22. Phosphate	9.3
9. Galacturonate	13.2	23. Arsenate	9.4
10. Bromate	7.8	24. Citrate	25.4
11. Chloride	4.0	25. Isocitrate	9.5
12. Nitrite	20.1	26. <i>cis</i> -Aconitate	11.4
13. Bromide	10.1	27. <i>trans</i> -Aconitate	11.8
14. Nitrate	11.5		

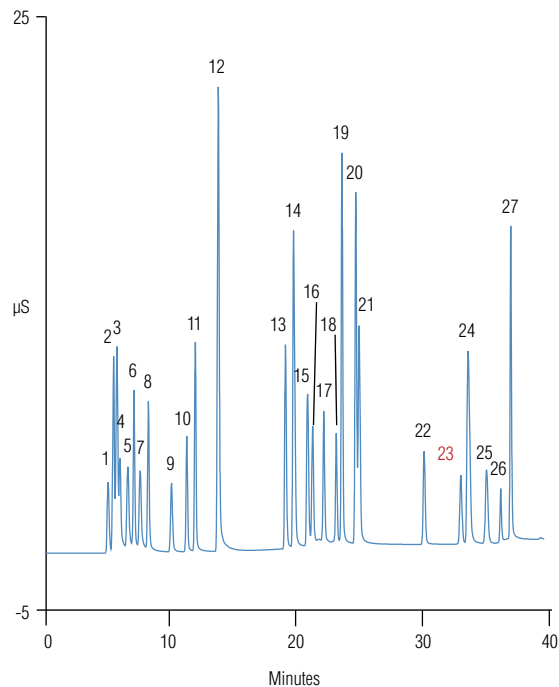


Figure 3. Mixed working standard.

To evaluate the linearity response to concentration, a calibration curve for arsenate was generated using replicate injections of 0.2 to 100 mg/L arsenate (Figure 4). Based on the signal to noise ratio (S/N), the arsenate LOD is 0.026 mg/L (S/N = 3, n = 5) and LOQ is 0.088 mg/L (S/N = 10, n = 5). The LOD and LOQ for total arsenic (As) is 0.014 and 0.047 mg/L respectively. These LODs/LOQs values approximate the EPA arsenic limit for drinking water of 0.010 ppm and well below the reported Lowest-Observed-Adverse-Effect Level (LOAEL) of 0.17 mg/L.<sup>4,5</sup>

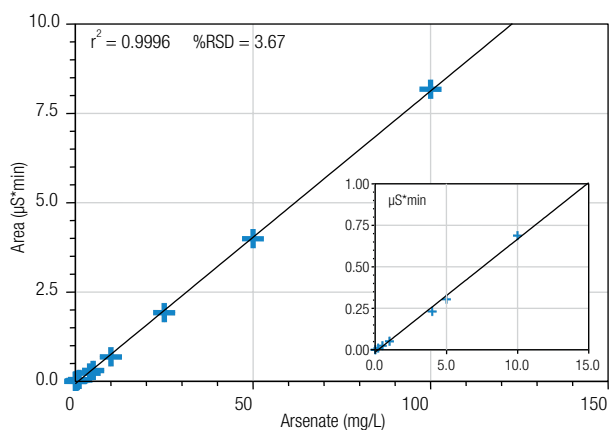


Figure 4. Calibration curve of arsenate from 0.2 to 100 mg/L.

During these experiments, a separate arsenite peak was not detected with this method. Interestingly, a corresponding concentration of arsenate was detected whenever arsenite was injected. To evaluate this hypothesis, 1 mg/L arsenate, 2 mg/L arsenite standards were tested neat (without treatment) and adjusted to pH 3 and pH 12. Figure 5 shows that freshly prepared arsenite samples were converted during the analysis to arsenate under acidic, neutral or basic conditions.

Researchers previously reported the conversion of As(III) to As(V), and vice versa.<sup>20</sup> A study by Segura et al., had shown that As(III) in wastewater is transformed into As(V) at most pHs.<sup>21</sup> More recently, another researcher reported that almost 100% of As(III) is converted to As(V) in tetramethylammonium hydroxide under basic conditions.<sup>22</sup> The eluent for this method, KOH, is also a strong base, which converts arsenite to arsenate during the analysis. Because both arsenite and arsenate are measured as arsenate, this is a suitable alternative method for total inorganic arsenic determinations.

Column: Dionex IonPac AS11-HC-4µm column, 0.4 × 250 mm  
 Instrument: Dionex ICS-5000+ HPIC system  
 Eluent Source: Dionex EGC-KOH cartridge (Capillary)  
 Gradient (KOH): 1.5–2 mM (-10–2 min), 2–8 mM (2–13 min), 8–28 mM (13–25 min), 28–35 mM (25–33 min), 35–65 mM (33–34 min), 65 mM (34–38 min).  
 Flow Rate: 0.015 mL/min  
 Inj. Volume: 0.40 µL  
 Column Temp.: 30 °C  
 IC Cube Temp.: 15 °C  
 Detection: Suppressed conductivity, Dionex ACES 300 Suppressor, AutoSuppression, recycle mode

Samples	Detected Arsenate (mg/L)
A: Water blank	–
B: 1 mg/L arsenate 2 mg/L arsenite	1.0
C: Sample B at pH = 3	2.1
D: Sample B at pH = 7	2.0
E: Sample B at pH = 12	2.0

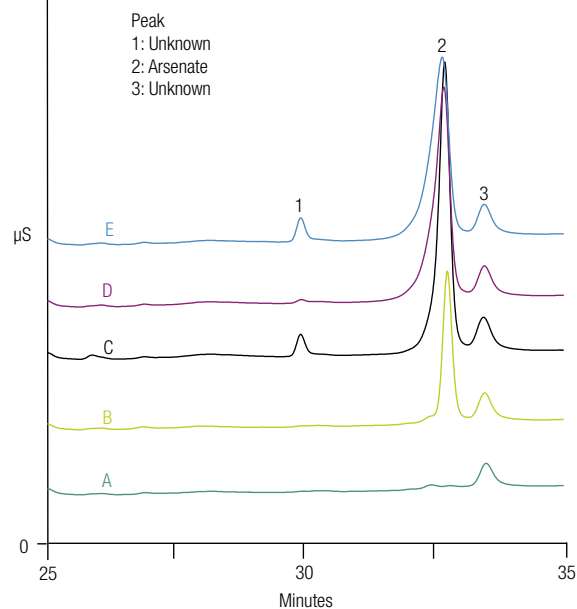


Figure 5. Arsenite detected as arsenate.

## Sample Analysis

The method was applied to six juice samples (grape, mango, cranberry juice cocktail) and three brands of apple juices. Figures 6–10 show the chromatograms of juice samples over-laid with water blank and arsenate spiked juice samples. The chromatograms show that only the apple juice samples had a peak at the arsenate retention time.

As demonstrated in these chromatograms, this method can characterize and quantify the organic acids, as well as, total inorganic arsenic in juices. Because the method detects most of the acids present in fruit juice, it can not only be used to detect low levels of inorganic arsenic, it can also be used to obtain an acid profile that is characteristic of a particular juice or wine.<sup>12,13</sup>

These chromatograms demonstrate the differences in composition between the various fruit juice samples. For example, apple juice contains 19 anions with succinate (or malate), galacturonate, and quinate being present at the highest concentrations (Figure 6).

Column: Dionex IonPac AS11-HC-4 $\mu$ m column, 0.4  $\times$  250 mm  
 Instrument: Dionex ICS-5000+ HPLC system  
 Eluent Source: Dionex EGC-KOH cartridge (Capillary)  
 Gradient (KOH): 1.5–2 mM (-10–2 min), 2–8 mM (2–13 min), 8–28 mM (13–25 min), 28–35 mM (25–33 min), 35–65 mM (33–34 min), 65 mM (34–38 min)  
 Flow Rate: 0.015 mL/min  
 Inj. Volume: 0.40  $\mu$ L  
 Column Temp.: 30  $^{\circ}$ C  
 IC Cube Temp.: 15  $^{\circ}$ C  
 Detection: Suppressed conductivity, Dionex ACES 300 Suppressor, AutoSuppression, recycle mode  
 Samples: A: Water  
 B: 50-fold dilution of apple juice Sample 1  
 C: 0.2 mg/L arsenate spiked Sample B  
 D: 0.5 mg/L arsenate spiked Sample B

Peak	mg/L	Peak	mg/L
1. Quinate	6.2	11. Malate/Succinate	73.5
2. Fluoride	1.0	12. Sulfate	1.5
3. Lactate	2.1	13. Oxalate	2.1
4. Acetate/Glycolate	3.7	14. Phosphate	3.9
5. Formate	2.6	15. Unknown	–
6. Pyruvate	0.4	16. Arsenate*	–
7. Galacturonate	15.7	17. Citrate	0.5
8. Chloride	0.3	18. Isocitrate	0.2
9. Nitrate	1.1	19. <i>trans</i> -Aconitate	0.3
10. Glutarate	0.3	20. Unknown	–

Note that the inset is a zoomed in view of the arsenate peak.

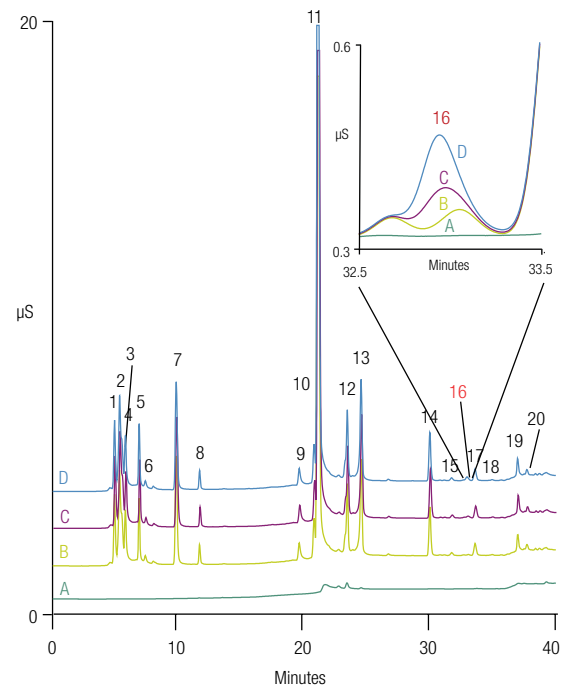


Figure 6. Inorganic anions, organic acids, and arsenate in a diluted apple juice sample.

Grape juice contains 18 anions with galacturonate, malonate (or tartrate), and succinate (or malate) being present at the highest concentrations (Figure 7). The mango juice sample in Figure 8, shows predominantly citrate with minor concentrations of other organic acids.

Column: Dionex IonPac AS11-HC-4 $\mu$ m column, 0.4  $\times$  250 mm  
 Instrument: Dionex ICS-5000+ HPIC system  
 Eluent Source: Dionex EGC-KOH cartridge (Capillary)  
 Gradient (KOH): 1.5–2 mM (-10–2 min), 2–8 mM (2–13 min), 8–28 mM (13–25 min), 28–35 mM (25–33 min), 35–65 mM (33–34 min), 65 mM (34–38 min)  
 Flow Rate: 0.015 mL/min  
 Inj. Volume: 0.40  $\mu$ L  
 Column Temp.: 30  $^{\circ}$ C  
 IC Cube Temp.: 15  $^{\circ}$ C  
 Detection: Suppressed conductivity, Dionex ACES 300 Suppressor, AutoSuppression, recycle mode

Samples:  
 A: Water  
 B: 50-fold dilution of a grape juice sample  
 C: 0.2 mg/L arsenate spiked Sample B  
 D: 0.5 mg/L arsenate spiked Sample B

Peak	mg/L	Peak	mg/L
1. Quinate	14.8	10. Malate/Succinate	39.7
2. Lactate	2.1	11. Malonate/Tartrate	63.6
3. Acetate	1.9	12. Sulfate	7.8
4. Formate	1.6	13. Oxalate	1.5
5. Pyruvate	0.4	14. Phosphate	9.2
6. Galacturonate	19.1	15. Unknown	na
7. Chloride	2.4	16. Arsenate*	
8. Nitrate	1.0	17. Citrate	3.9
9. Glutarate	na	18. Isocitrate	0.7
		19. trans-Aconitate	na

Note that the inset is a zoomed in view of the arsenate peak.

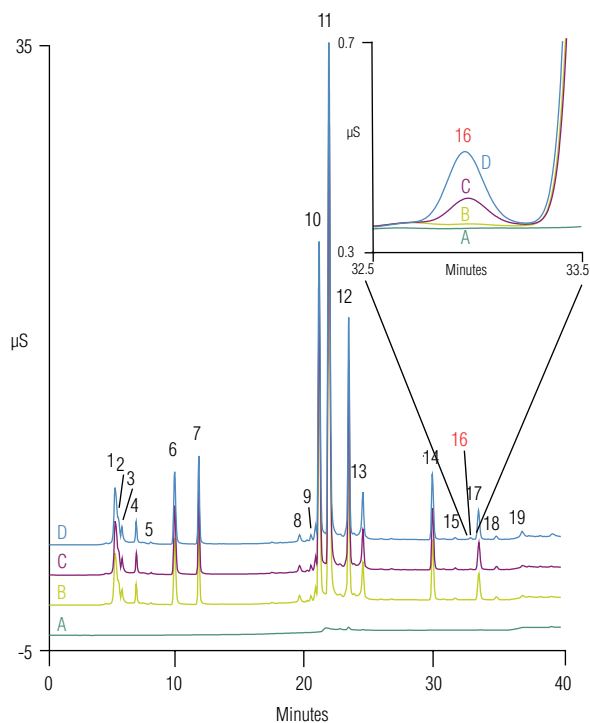


Figure 7. Inorganic anions, organic acids, and arsenate in a diluted grape juice sample.

Column: Dionex IonPac AS11-HC-4 $\mu$ m column, 0.4  $\times$  250 mm  
 Instrument: Dionex ICS-5000+ HPIC system  
 Eluent Source: Dionex EGC-KOH cartridge (Capillary)  
 Gradient (KOH): 1.5–2 mM (-10–2 min), 2–8 mM (2–13 min), 8–28 mM (13–25 min), 28–35 mM (25–33 min), 35–65 mM (33–34 min), 65 mM (34–38 min)  
 Flow Rate: 0.015 mL/min  
 Inj. Volume: 0.40  $\mu$ L  
 Column Temp.: 30  $^{\circ}$ C  
 IC Cube Temp.: 15  $^{\circ}$ C  
 Detection: Suppressed conductivity, Dionex ACES 300 Suppressor, AutoSuppression, recycle mode

Samples:  
 A: Water  
 B: 50-fold dilution of a mango juice sample  
 C: 0.2 mg/L arsenate spiked into Sample B  
 D: 0.5 mg/L arsenate spiked into Sample B

Peak	mg/L	Peak	mg/L
1. Quinate	2.3	10. Maleate	0.3
2. Lactate	2.1	11. Sulfate	0.6
3. Acetate	2.2	12. Oxalate	0.2
4. Formate	0.5	13. Phosphate	1.0
5. Galacturonate	0.6	14. Arsenate*	
6. Chloride	0.5	15. Citrate	25.4
7. Nitrate	0.7	16. Isocitrate	0.3
8. Malate/Succinate	1.1		
9. Malonate/Tartrate	0.1		

Note that the inset is a zoomed in view of the arsenate peak.

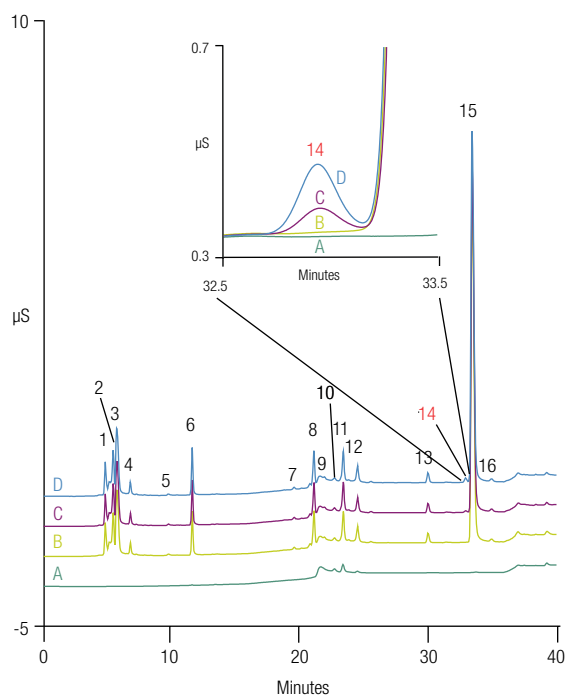


Figure 8. Inorganic anions, organic acids, and arsenate in a diluted mango juice sample.

Cranberry cocktail juice also contains 18 anions, but the anions at the highest concentration are quinate, succinate (or malate) and citrate (Figure 9).

Column: Dionex IonPac AS11-HC-4 $\mu$ m column, 0.4  $\times$  250 mm  
 Instrument: Dionex ICS-5000<sup>+</sup> HPIC system  
 Eluent Source: Dionex EGC-KOH cartridge (Capillary)  
 Gradient (KOH): 1.5–2 mM (-10–2 min), 2–8 mM (2–13 min),  
 8–28 mM (13–25 min), 28–35 mM (25–33 min),  
 35–65 mM (33–34 min), 65 mM (34–38 min)  
 Flow Rate: 0.015 mL/min  
 Inj. Volume: 0.40  $\mu$ L  
 Column Temp.: 30  $^{\circ}$ C  
 IC Cube Temp.: 15  $^{\circ}$ C  
 Detection: Suppressed conductivity, Dionex ACES 300 Suppressor,  
 AutoSuppression, recycle mode  
 Samples:  
 A: Water  
 B: 50-fold dilution of a cranberry cocktail juice sample  
 C: 0.2 mg/L arsenate spiked into Sample B  
 D: 0.5 mg/L arsenate spiked into Sample B

Peak	mg/L	Peak	mg/L
1. Quinate	38.8	10. Malate/Succinate	43.4
2. Fluoride	0.2	11. Maleate	0.8
3. Lactate	0.4	12. Sulfate	0.4
4. Acetate	0.6	13. Oxalate	—
5. Formate	0.2	14. Phosphate	0.8
6. Pyruvate	0.3	15. Unknown	na
7. Galacturonate	7.5	16. Arsenate*	
8. Chloride	0.3	17. Citrate	56.0
9. Nitrate	0.7	18. Isocitrate	0.4
		19. trans-Aconitate	0.4

Note that the inset is a zoomed in view of the arsenate peak.

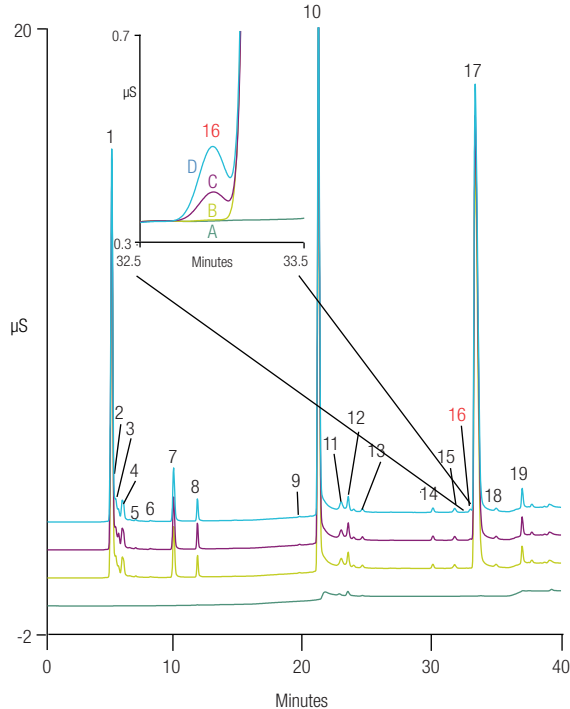


Figure 9. Inorganic anions, organic acids, and arsenate in a diluted cranberry cocktail juice.

The profile of juices can be used to assess whether an expensive juice has been illegally adulterated with a lower cost juice such as cranberry with apple juice (Figure 10). Because the peak ratio of malate/succinate to quinate is high for apple juice (about 12 to 1) and low for cranberry juice (about 1 to 1), adding apple juice into cranberry juice is easily identified with this peak ratio.

Column: Dionex IonPac AS11-HC-4 $\mu$ m column, 0.4  $\times$  250 mm  
 Instrument: Dionex ICS-5000<sup>+</sup> HPIC system  
 Eluent Source: Dionex EGC-KOH cartridge (Capillary)  
 Gradient (KOH): 1.5–2 mM (-10–2 min), 2–8 mM (2–13 min),  
 8–28 mM (13–25 min), 28–35 mM (25–33 min),  
 35–65 mM (33–34 min), 65 mM (34–38 min).  
 Flow Rate: 0.015 mL/min  
 Inj. Volume: 0.40  $\mu$ L  
 Column Temp.: 30  $^{\circ}$ C  
 IC Cube Temp.: 15  $^{\circ}$ C  
 Detection: Suppressed conductivity, Dionex ACES 300 Suppressor,  
 AutoSuppression, recycle mode  
 Samples:  
 A: Water  
 B: 1 mg/L arsenate in water  
 C: 50-fold dilution of apple juice Sample 1  
 D: 0.50 mg/L arsenate spiked Sample C  
 E: 50-fold dilution of apple juice Sample 2  
 F: 50-fold dilution of apple juice Sample 3

Note that the inset is a zoomed in view of the arsenate peak.

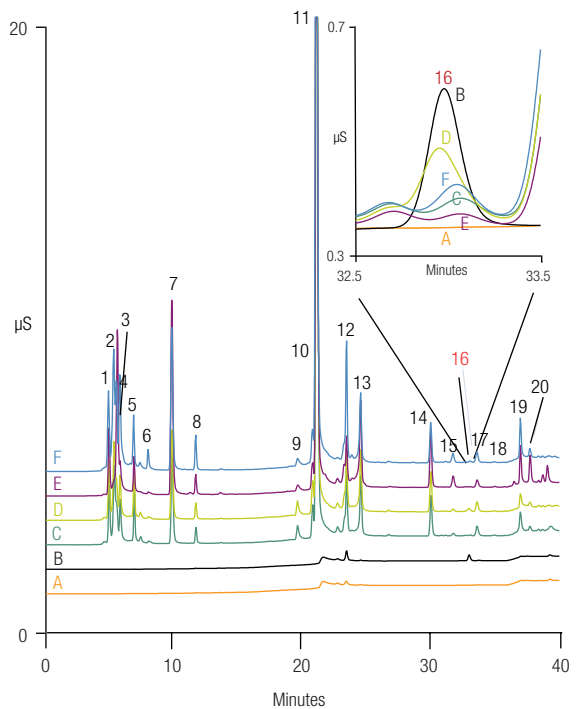


Figure 10. Inorganic anions, organic acids, and arsenate in a diluted apple juice samples.



## Conclusion

The high-pressure Dionex ICS-5000<sup>+</sup> HPIC IC system and the newly introduced Thermo Scientific Dionex ICS-4000 capillary HPIC system provide a solution for simple juice analysis.

This technical note demonstrates that:

- High-pressure capillary IC with suppressed conductivity detection (CD) provides a sensitive method to detect and quantify arsenite (As(III)) and arsenate (As(V)) as total inorganic arsenic, as well as characterizing organic acids
- With an arsenate LOD of 0.026 mg/L and LOQ of 0.088 mg/L, this method can be used to estimate the total inorganic arsenic (As) in juice at a LOD of 0.014 mg/L and LOQ of 0.047 mg/L, which is well below the EPA reported LOAEL of 0.17 mg/L

## References

1. Letters from the FDA to the Dr.Oz Show Regarding Apple juice and Arsenic. [Online] <http://www.fda.gov/Food/ResourcesForYou/Consumers/ucm271746.htm> (accessed Feb. 14, 2014).
2. Arsenic in Apple Juice. [Online] <http://www.fda.gov/Food/FoodborneIllnessContaminants/Metals/ucm280209.htm> (accessed Feb. 14, 2014).
3. FDA Widens Look at Arsenic in Apple Juice. [Online] <http://www.fda.gov/ForConsumers/ConsumerUpdates/ucm283235.htm> (accessed Feb. 14, 2014).
4. Arsenic in Drinking Water. [Online] <http://water.epa.gov/lawsregs/rulesregs/sdwa/arsenic/index.cfm> (accessed Feb. 14, 2014).
5. Arsenic, inorganic (CASRN7440-38-2). [Online] <http://www.epa.gov/iris/subst/0278.htm> (accessed Feb. 14, 2014).
6. Arsenic. [Online] <http://www.hc-sc.gc.ca/fn-an/securit/chem-chim/envIRON/arsenic-eng.php> (accessed Feb. 14, 2014).
7. EPA-815-R-00-010, *Analytical Methods Support Document For Arsenic In Drinking Water*. 1999. [Online] <http://www.epa.gov/ogwdw/arsenic/pdfs/methods.pdf> (accessed Feb. 14, 2014).
8. Thermo Fisher Scientific. Application Note 43099, *IC-ICP-MS speciation analysis of As in apple juice using the Thermo Scientific iCAP Q ICP-MS*, AN43099\_E 03/12C, Sunnyvale, CA, 1992.
9. Thermo Fisher Scientific, Application Update 154, AU 154 *Determination of Inorganic Anions in Wastewater in Environmental Waters Using a Hydroxide-Selective Column*, LPN 1539, Sunnyvale, CA, 2003.
10. Thermo Fisher Scientific, Application Note 116, AN 116 *Quantification of Anions in Pharmaceuticals*, LPN 0924, Sunnyvale, CA, 2002.
11. Thermo Fisher Scientific, Application Note 153, AN 153 *Monitoring for Trace Anion Contamination in the Extracts of Electronic Components*, LPN 1527, Sunnyvale, CA, 2003.
12. Thermo Fisher Scientific, Application Note 143, AN 143 *Determination of Organic Acids in Fruit Juices*, LPN 1415, Sunnyvale, CA, 2003.
13. Thermo Fisher Scientific, Application Note 273, AN 273 *Higher Resolution Separation of Organic Acids and Common Inorganic Anions in Wine*, LPN 2727, Sunnyvale, CA, 2011.
14. Thermo Fisher Scientific. Technical Note 113, TN 113 *Practical Guidance for Using Capillary Anion Chromatography*, LPN 3043, Sunnyvale, CA, 2012.
15. Thermo Fisher Scientific. Dionex ICS-5000<sup>+</sup> Installation Manual, Doc No. 065447, Sunnyvale, CA, 2012.
16. Thermo Fisher Scientific. Technical Note 131, TN 131 *Configuring High-Pressure Capillary IC on the Modular IC System*, Document No. TN70352\_E 12/12S, Sunnyvale, CA, 2013.
17. Thermo Fisher Scientific. Product Manual for the Continuously Regenerated Trap Column (CR-TC), Doc No. 031910, Sunnyvale, CA, 2010.
18. Thermo Fisher Scientific. Product Manual for CES 300 Suppressors, Doc No. 065386, Sunnyvale, CA, 2010.
19. Thermo Fisher Scientific. Dionex AS-AP Operator's Manual, Doc No. 065361, Sunnyvale, CA, 2012.
20. K.A. Francesconi, D. Kuehnelt, *Analyst*, 2004, 129, 373–395.
21. M. Segura, J. Munoz, Y. Madrid and C. Camara, *Anal. Bioanal. Chem.*, 2002, 374, 513–519.
22. A Comparison of Arsenic Speciation Methods for Biological Tissues. [Online] <http://nemc.us/docs/2012/presentations/MonPM-Metallic-Ugrai-8-6-12.pdf> (accessed Feb. 14, 2014).

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