

# Determination of Iodide in Saline Matrices

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## INTRODUCTION

Iodine deficiency affects thyroid hormone production and leads to developmental diseases, goiter, and paralysis.<sup>1</sup> Table salt is routinely iodized to prevent these diseases and to promote public health. Iodization levels vary by country and can range from 5–100 mg iodine per kg of salt.<sup>2-3</sup> Two methods for determining iodide are presented in this poster. The first method uses an IonPac® AS20 for separation with conductivity detection followed by UV detection. No sample preparation is required.<sup>4</sup> A second method, allowing simultaneous determination of iodate and iodide, was developed using the Acclaim® Mixed-Mode WAX-1 column and UV detection. Only moderate sample dilution is necessary for iodate determination.<sup>5</sup>

## EXPERIMENTAL

### (Method 1)

A Dionex ICS-3000 RFIC™ system equipped with:  
 EluGen® EGC II KOH cartridge  
 IonPac AS20 (2 × 250 mm) and AG20 (2 × 50 mm)  
 ASRS® 300 (2 mm) suppressor operating at 28 mA in external water mode  
 VWD-3400 UV detector

### (Method 2)

A Dionex UltiMate® 3000 system equipped with:  
 SRD-3200 Solvent Rack  
 HPG-3200M Pump  
 WPS-3000TSL Micro Autosampler  
 TCC-3200 Column Compartment  
 PDA-3000 Detector  
 Acclaim Mixed-Mode WAX-1 Column, 2.1 × 150 mm

The Chromeleon® Chromatography Data System software was used for system control and data processing.

## RESULTS

### (Method 1)

Table 1. Linearity, LOD, LOQ, and Precision for Iodide Determination by UV and CD							
Detection	RT (min)	Range (µg/L)	Correlation Coefficient (r <sup>2</sup> )	LOD* (µg/L)	LOQ* (µg/L)	RT Precision (RSD)**	Peak Area Precision (RSD)**
Conductivity	19.33	50–250	0.9967	15	50	0.013	1.67
UV	19.43	50–250	0.9962	15	50	0.032	1.34

\* LOQ and LOD are highly dependent on the noise the day they are performed. The LOD and LOQ are concentrations that resulted in peaks during three days of testing that were an average of 3x and 10x the noise, respectively.

\*\* Seven injections of 100 µg/L iodide standard.

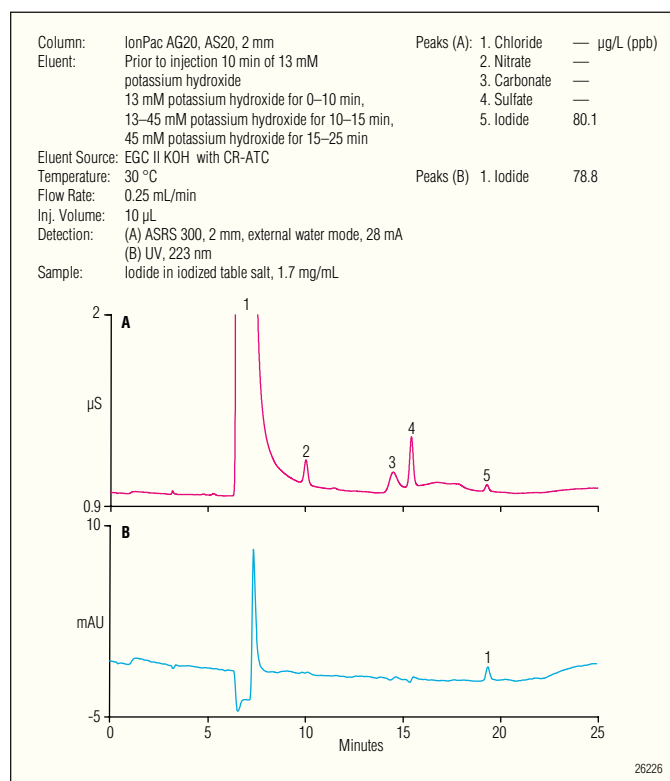


Figure 1. Separation of iodide in iodized table salt.

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UV detection provides a clear advantage with samples containing high concentrations of carbonate and sulfate. Iodide is underestimated by conductivity detection in these samples.

## RESULTS (Method 2)

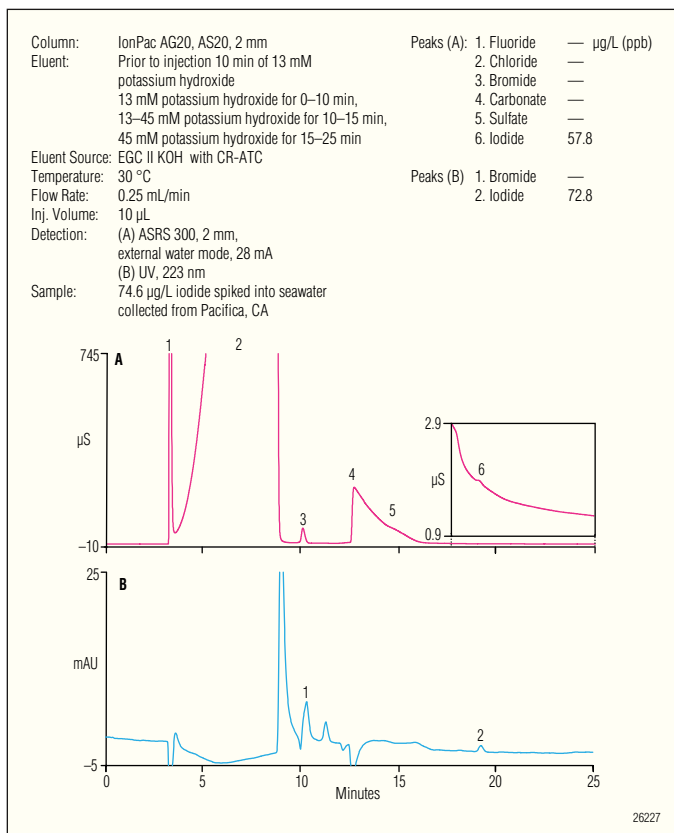


Figure 2. Separation of iodide in seawater from Pacifica, CA.

Iodide recoveries by UV detection using the IonPac AS20 column are excellent, showing method accuracy (Table 2).

Sample	Amount Found in Spiked Sample (µg/L)	Amount Added (µg/L)	Recovery (%)
Simulated seawater	48.2 ± 2.0	49.5	97 ± 4.1
	142 ± 1.2	141	101 ± 0.9
	252 ± 1.5	244	103 ± 0.6
Synthetic sea salt	201 ± 0.8	96.6	94 ± 0.4
Table salt, fresh	189 ± 1.0	99.1	99 ± 0.5
Seawater, Half Moon Bay, CA	70.5 ± 0.8	73.2	96 ± 1.2
Seawater, Pacifica, CA	72.4 ± 0.7	74.6	97 ± 1.0

Analyte	RT (min)	Range (µg/L)	Correlation Coefficient (r <sup>2</sup> )	LOD (µg/L)	LOQ (µg/L)	RT Precision (RSD)*	Peak Area Precision (RSD)*
Iodate	5.61	500-7600	0.9994	60	200	0.05	0.95
Iodide	12.54	10-250	0.9985	3.3	9.9	0.04	0.69

\* Precision data determined from 7 injections of 100 µg/L iodide and 1.5 mg/L iodate in deionized water.

The Acclaim Mixed-Mode WAX-1 column allows simultaneous determination of iodide and iodate.

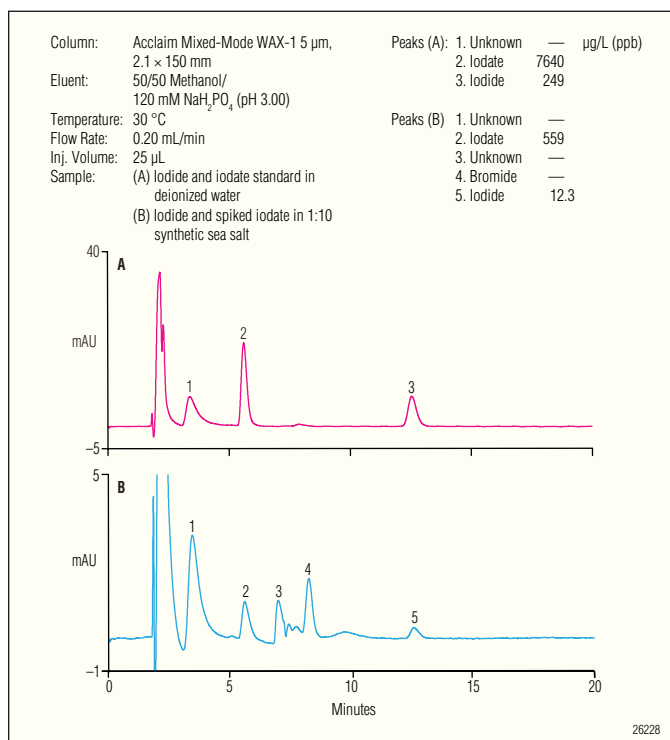


Figure 3. Separation of iodide and iodate standard in deionized water and synthetic sea salt.

Iodide is well resolved with direct injection of saline samples.

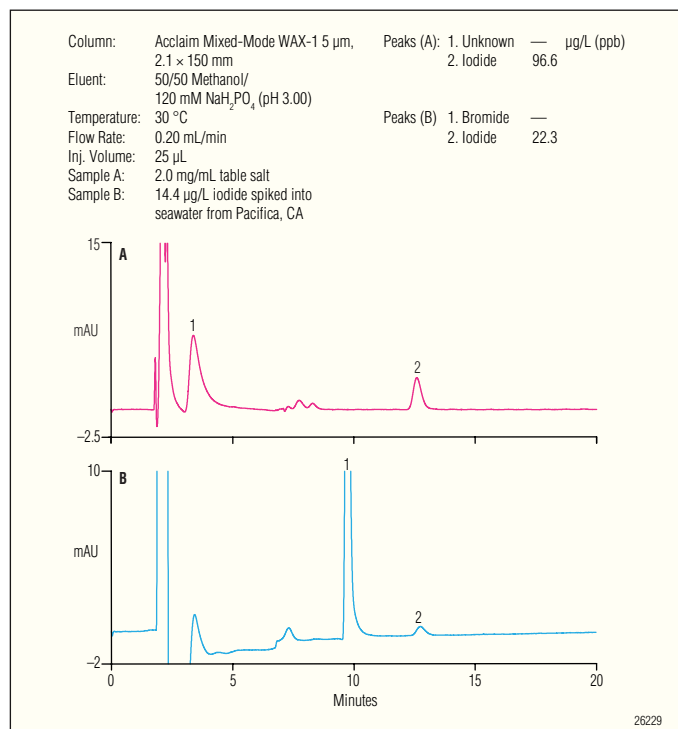


Figure 4. Separation of iodide in table salt and seawater on the Mixed Mode WAX-1 column.

Table 4. Recoveries of Iodate and Iodide from Saline Samples, Triplicate Injections				
Sample	Analyte	Amount Found in Spiked Sample ( $\mu$ g/L)	Amount Added ( $\mu$ g/L)	Recovery (%)
Simulated seawater	Iodate*	530 $\pm$ 6.0	490	108 $\pm$ 1.2
	Iodide	52 $\pm$ 0.4	50	104 $\pm$ 0.8
		160 $\pm$ 0.8	141	113 $\pm$ 0.6
Synthetic sea salt	Iodate*	550 $\pm$ 5.0	520	104 $\pm$ 0.9
	Iodide	197 $\pm$ 2.7	96.6	93 $\pm$ 1.3
Table salt, fresh	Iodide	196 $\pm$ 1.8	99.1	103 $\pm$ 0.9
Seawater, Pacifica, CA	Iodide	21.5 $\pm$ 0.7	14.4	101 $\pm$ 3.3
Seawater, Half Moon Bay, CA	Iodide	20.5 $\pm$ 1.6	14.0	93 $\pm$ 7.3

\*Recoveries of iodate were from samples that had been diluted 1:10 in DI water.

Both methods perform well when analyzing samples.

Table 5. Determination of Iodide in Saline Samples by UV Detection, Triplicate Injections					
Sample	RT (min)	RT Precision (RSD)	Peak Area Precision (RSD)	Amount Found ( $\mu$ g/L)	Amount in Dry Salt ( $\mu$ g/g)
Method 1					
Synthetic sea salt (30.4 mg/mL)	19.36	0.05	0.524	108 $\pm$ 0.6	3.6 $\pm$ 0.02
Fresh table salt (1.98 mg/mL)	19.43	<0.01	1.24	92.4 $\pm$ 1.2	46.7 $\pm$ 0.7
Method 2					
Synthetic sea salt (30.4 mg/mL)	12.63	0.22	1.18	122 $\pm$ 1.4	4.0 $\pm$ 0.05
Fresh table salt (1.68 mg/mL)	12.74	0.21	0.11	80.3 $\pm$ 0.1	47.8 $\pm$ 0.05
Aged table salt (2.74 mg/mL)	12.67	0.06	0.96	119 $\pm$ 1.1	43.4 $\pm$ 0.4
Seawater (Pacifica, CA)	12.85	0.51	6.62	9.3 $\pm$ 0.6	—
Seawater (Half Moon Bay, CA)	12.66	0.24	6.69	7.6 $\pm$ 0.5	—

## CONCLUSION

With no sample dilution, iodide can be accurately determined in seawater and other saline samples with recoveries ranging from 94–103% on the IonPac AS20 column (Figure 2 and Table 2). A second method, allowing simultaneous determination of iodate and iodide, was developed using the Acclaim Mixed-Mode WAX-1 column and UV detection (Figure 3). The iodide amount determined in table salt was 46.7  $\mu$ g/g of salt using the IonPac AS20 column and 47.8  $\mu$ g/g using the Acclaim Mixed-Mode WAX-1 column, showing equivalent iodide quantification for the two methods when analyzing saline solutions.

## REFERENCES

- 1) *Iodine Status Worldwide*: WHO Global Database on Iodine Deficiency, de Benoist, B.; Anderson, M.; Egli, I.; Takkouche, B.; Allen, H.; eds. Department of Nutrition for Health and Development, World Health Organization, Geneva, 2004.
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- 4) Dionex Corporation. Application Note 239. Determination of Iodide in Seawater and other Saline Matrices using a Reagent-Free™ Ion Chromatography System with Suppressed Conductivity and UV Detection. LPN 2334, Dionex Corporation, Sunnyvale, CA. **2009**.
- 5) Dionex Corporation. Application Note 236. Determination of Iodide and Iodate in Seawater and Iodized Table Salt by HPLC with UV Detection. LPN 2312, Dionex Corporation, Sunnyvale, CA. **2009**.

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