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Determination of Iodide in a Nutritional Supplement Using an Acclaim Mixed-Mode WAX-1 Column

Iodide is an essential micronutrient. The use of iodinated salt in the diet is the most frequent way of obtaining additional dietary iodide. In addition to ion chromatography (IC), which is a commonly used method for determining iodide, there is still a need for an HPLC method for use with some samples.¹ Because of very weak retention of iodide on a conventional reversed-phase (RP) C18 column, two alternate RP approaches to determine iodide have been used. These alternate approaches are to add an ion-pair reagent to the mobile phase² and/or to perform precolumn derivatization of iodide.³ A simpler approach is to use an RP stationary phase that also contains ion-exchange functional groups for simultaneous separation of neutral and ionic compounds. The Acclaim[®] Mixed-Mode WAX-1, a column that combines anion-exchange and RP properties, was used in AN 236 to determine iodide and iodate in seawater and iodized table salt.⁴

The work shown here describes an efficient method for determining iodide in a nutritional supplement (tablet) using a method similar to AN 236. The determination was completed on an Acclaim Mixed-Mode WAX-1 column (3.0 × 150 mm, 3 μm) using a mobile phase. The mobile phase consists of a phosphate buffer (pH 6.3, adjusted by adding 100 mM K₂HPO₄ to a 500 mL of 70 mM KH₂PO₄ solution) and acetonitrile mixed by the HPLC pump (72:28, v/v), with a detection wavelength of 220 nm. Figure 1 shows that a nutritional supplement had an iodide content of 88 μg/g, which is consistent with the labeled amount (100 μg/g).

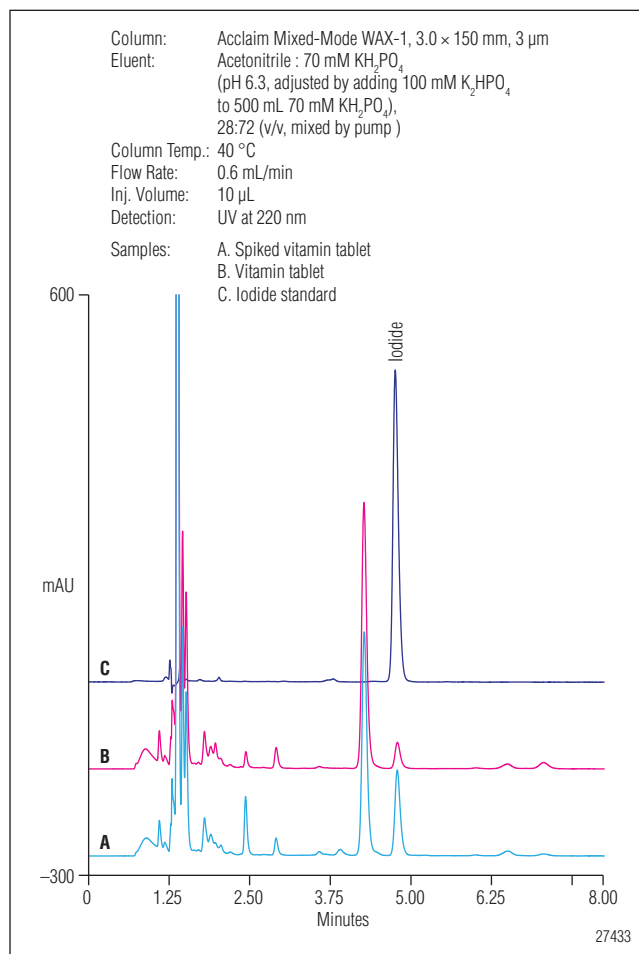


Figure 1. Overlay of chromatograms of A) spiked sample, B) sample, and C) iodide standard.

EQUIPMENT

Dionex UltiMate® 3000 RSLC system, including:
HPG-3400RS Pump, WPS-3000RS Autosampler,
TCC-3000RS Thermostatted Column Compartment,
DAD-3000RS Diode Array Detector
Chromeleon® 6.80 SR7 Chromatography Data System

SAMPLE PREPARATION

Put 0.100 g of ground vitamin tablet into a 10 mL centrifuge tube, add 8 mL water, and then place in an ultrasonic bath for 15 min. Samples should be prepared just prior to analysis to prevent loss of iodide by oxidation. Prior to injection, filter the solution through a 0.2 µm filter (Millex®-HN).

REFERENCES

1. Dionex Corporation, *Determination of Iodide in Seawater and Other Saline Matrices Using a Reagent-Free Ion Chromatography System with Suppressed Conductivity and UV Detections*. Application Note 239, LPN 2334, 2009, Sunnyvale, CA.
2. Rendl, J.; Seybold, S.; Borner, W. Urinary Iodide Determined by Paired-Ion Reversed-Phase HPLC with Electrochemical Detection, *Clinical Chemistry*, **1994**, *40*, 908–913.
3. Verma, K.K.; Jain, A.; Verma, A. Determination of Iodide by High-Performance Liquid Chromatography after Precolumn Derivatization, *Anal. Chem.*, **1992**, *64*, 1484–1489.
4. Dionex Corporation, *Determination of Iodide and Iodate in Seawater and Iodized Table Salt by HPLC with UV Detection*. Application Note 236, LPN 2312, 2009, Sunnyvale, CA.

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LPN 2538 PDF 6/10
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