

Application Note 14A

Rapidly Analyze Sodium in Foods, Using Solid Phase Extraction and Cation-Exchange HPLC

Sodium in canned corn and tomato soup was monitored using cation-exchange high performance liquid chromatography. Samples were cleaned using Supelclean LC-18 solid phase extraction tubes. The tube effectively retained interfering food sample components, while allowing sodium ions to elute. Sodium was monitored in the eluate using a SUPELCOSIL LC-SCX column and conductivity detection. Response was linear over a sodium concentration range of 0.1-2mg/mL, and there was good correlation between the food label claims and the measured sodium content.

Key Words

- sodium
- solid phase extraction
- foods

Cation-exchange high performance liquid chromatography is an effective analytical technique for monitoring sodium ions (1). However, complex sample matrices — such as food products — must be cleaned before HPLC analysis or they will clog the column. (Simple filtration removes particulate matter, but not other matrix components that could interfere with sodium analysis.) Supelclean™ LC-18 reversed phase solid phase extraction tubes retain the bulk of the potentially interfering food sample components, while allowing sodium ions to elute. The extraction method is simple, rapid, and reliable.

Cans of regular and low sodium whole-kernel corn were drained and the canning liquids saved. Three-milliliter Supelclean LC-18 solid phase extraction tubes were conditioned with 3.0mL of methanol, and dried under nitrogen. (Up to 12 or 24 tubes can be conditioned simultaneously, using a Visiprep™ Solid Phase Extraction Vacuum Manifold equipped with a Visidry™ Drying Attachment.) Particulate and dissolved organic matter was removed from the corn liquid by passing the sample (about 5mL) through the tube. The collected eluate was analyzed without further treatment.

Similarly, condensed regular and low sodium tomato soups were diluted according to the serving recommendations (1:1), using mobile phase (0.05M citric acid). One-milliliter Supelclean LC-18 SPE tubes were conditioned with 1.0mL of methanol and dried under nitrogen. Even after dilution, the highly viscous and particulate soups clogged the SPE tubes. This problem was alleviated by inserting a plug of silanized glass wool into the SPE tube (after conditioning), to trap sample particulate matter. Soup samples (about 3mL) were then passed through the tubes successfully, and the eluate was analyzed.

Cation exchange analyses were carried out on a 25cm x 4.6mm SUPELCOSIL™ LC-SCX column, under isocratic conditions, using a

0.05M citric acid mobile phase (pH 2.4). A column temperature of 35°C ensured a stable baseline, and triplicate injections of 1µL (regular product) or 10µL (low sodium product) were made. Sodium was detected by conductivity.

Results for triplicate injections of the SPE purified food samples are summarized in Table 1. Response was linear for the range of sodium standards used (0.1-2mg/mL), and there was good correlation between the food label claims and the measured sodium content. Chromatograms of HPLC/conductivity analyses for sodium in the corn liquids and tomato soup samples are shown in Figure A.

Figure A . Sodium in Corn Canning Liquids and Tomato Soup

Column: SUPELCOSIL LC-SCX, 25cm x 4.6mm, 5µm particles
 Cat. No.: 58997
 Temp.: 35°C
 Mobile Phase: 0.05M citric acid, pH 2.4
 Flow Rate: 2.0mL/min
 Det.: conductivity
 Inj.: Whole corn canning liquids; tomato soup concentrates diluted with 1:1 mobile phase

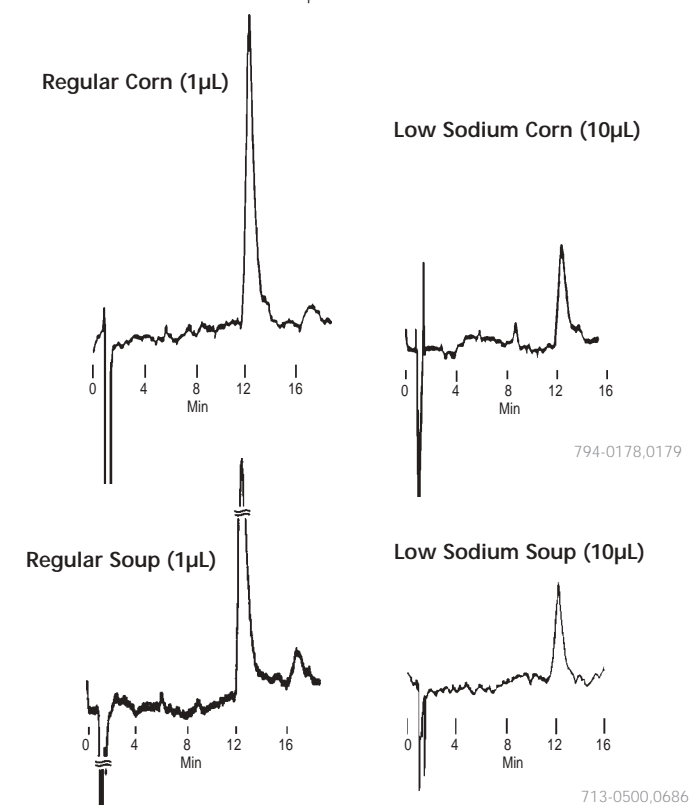


Table 1 — Sodium Accurately Monitored in Corn Canning Liquids and Tomato Soup

Food Product	Label Claim (mgNa/mL)	X	Injection Volume (µL)	Total Na Expected (µg)	Na Measured (µg) (mean, N=3)
Corn Liquids					
Regular	3.12	1		3.12	3.09
Low Sodium	0.10	10		1.00	1.00
Tomato Soup					
Regular	4.58	1		4.58	4.67
Low Sodium	0.13	10		1.30	1.28

A conductivity detector — used in combination with an ion exchange HPLC system — will be sensitive to column equilibration and to changes in mobile phase makeup and column temperature. About 50-100mL of mobile phase was needed to achieve column equilibration. We found it almost impossible to obtain a stable baseline at ambient temperature, but baselines stabilized at temperatures above 30°C.

The SPE method used to purify these food samples effectively retains particulate and organic matter, while passing sodium, unretained, in the breakthrough volume of the packing bed. Application of the method may be complicated by sample viscosity. While corn liquids passed freely through the SPE tubes, tomato soup required dilution to prevent back pressure from becoming undesirably high. Sample dilution can, in some cases, affect sodium detection. For many food samples, however, this method should be very useful for separating particulate material and organic components from sodium, when monitoring this ion.

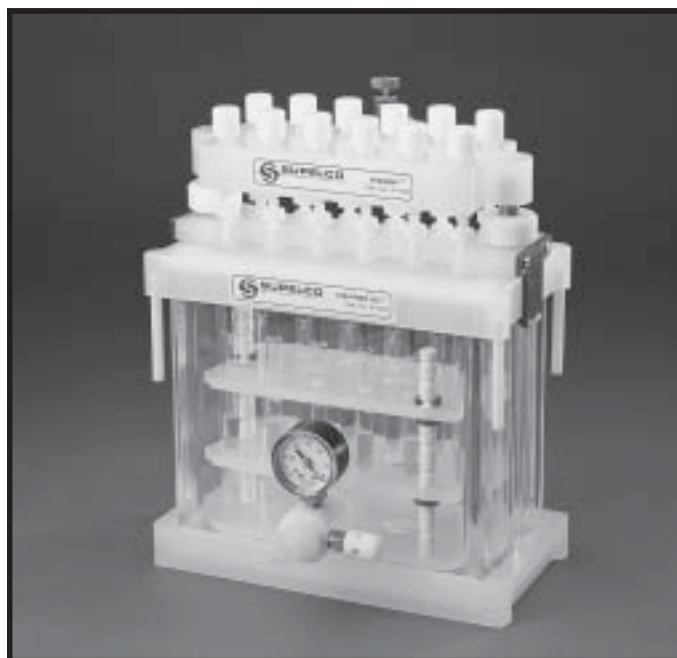
Reference

1. Smith, R.L. and D.J. Pietrzyk, *Anal. Chem.*, 56, 610 (1984).
Reference not available from Supelco.

Trademarks

Supelclean, SUPELCOSIL, Visidry, Visiprep — Sigma-Aldrich Co.

12-Port Visiprep DL Manifold Fitted with Visidry Drying Attachment



995-0140

Ordering Information:

Description	Cat. No.
Supelclean LC-18 Solid Phase Extraction Tubes	
1mL, pk. of 100	57002-U
3mL, pk. of 50	57012
12-Port Visiprep Solid Phase Extraction Vacuum Manifold	57030-U
Visidry Vacuum Manifold Drying Attachment	57100-U
Silanized Glass Wool	
50g	20411
250g	20410
SUPELCOSIL LC-SCX Column	
25cm x 4.6mm, 5µm packing	58997

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