

6 Injection Mode: Hydrostotic 10 cm for 30 sec Detection: UV @ 185nm Time Constant: 0.3 sec.

The electropherogram above shows the separation of a group of metal cations by capillary ion analysis (CIA), with indirect UV absorbance detection at 185 nm. With all other conditions identical, improved sensitivity is obtained by employing a 185 nm lamp and filter kit.

# The Versatility of Capillary Ion Analysis

Minutes

Capillary Ion Analysis (CIA) is a capillary electrophoretic technique optimized for the analysis of small molecular weight inorganic and organic ions. Sensitive detection is based predominantly on indirect UV since the majority of the ions lack specific chromophores. Rapid separations are a result of directing the electroosmotic flow towards the detector in combination with an appropriately mobile background electrolyte co-ion. Up to 1,000,000 theoretical plates can be achieved through the proper selection of electrolyte co-ion mobility along with care and consideration for field strength and capillary dimensions. Selectivity of separations can easily be predicted with the aid of readily available ionic equivalent conductances

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Waters patented Capillary Ion Analysis technology has already amassed numerous references, and interest in this technology is increasing. For this reason, we have produced a detailed review article on the versatility Capillary Ion Technology.

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Photodiode Array (PDA) Detector\*. Even though mass spectrometry is relied upon for positive confirmation, PDA detection supplies important qualitative information such as peak purity and spectral matching.

#### Sample Preparation:

Solid Phase Extraction using Sep-Pak Cartridges has been successfully used for extracting veterinary drug residues since the early 1980s. Dr. Boison suggests the use of solid phase extraction is critical for trace level HPLC analysis of penicillins in animal tissue. A Waters Sep-Pak Plus tC<sub>18</sub> cartridge was found to give excellent recoveries of penicillin G and  $V^2$ . The sorbent used in this cartridge,  $tC_{18}$ , is a specially developed, high reproducibility, high carbon load packing material based on Waters state-of-theart trifunctional bonding chemistry process.

#### Sample Protocol A:

Bovine tissues are extracted with water and proteins and are precipitated with acid. The extract supernatant is then trace enriched on the Sep-Pak tC<sub>18</sub> cartridge. Figure 1 and Table 1 show a comparison of the recoveries from several different solid phase extraction products as well as the HPLC analysis.

### II.Tetracyclines

John Walsh and associates at the Regional Veterinary Laboratory, Department of Agriculture, Victoria, Australia used Waters Sep-Pak Environmental cartridges to measure tetracyclines in beef and pork<sup>2</sup>. The aim of this work was to produce consistent peak areas in tissues spiked at the 100 ng/g level with a reasonable turn-around time. The Environmental Sep-Pak Plus cartridges contain over twice the amount of monofunctional C., sorbent as the standard "short body" Sep-Pak Plus Cartridges. This cartridge provides the additional capacity needed to meet the required sensitivity.\*

For ordering information, see page 30. For more information on Waters sample prep products, please check box number 9 on the business reply card and return today.

Bovine and porcine muscle is homogenized in EDTA-McIlvaine buffer, centrifuged and proteins are precipitated with acid. The extract is then cleaned up and concentrated on the Sep-Pak Environmental Cartridge.

## An Excellent Aid for Your Methods Development Effort

The Sep-Pak Cartridge Applications Bibliography can be of significant help in developing your solid phase extraction protocols. It contains references to over 2,000 papers published by researchers around the world using Sep-Pak cartridges to solve their sample preparation problems. It is very convenient to use with three separate indices which include:

- Compound (over 5,700 compounds)
- Sample Matrix (1,000 matrices)
- Author (6,700 authors)

These indices help to save time in finding references closely related to your applications.

\* Note: Both monofunctional and trifunctional  $C_{18}$  sorbents are now available in even larger size cartridges with sorbent mass up to 10 grams per cartridge. For more information on large size cartridges, see the highlighted section below and ordering information on page 30.

#### References

1, JAOAC 74 (1991) 497–501 2, Journal of Chromatography, 1992 In Press.

# Large Volume Sep-Pak® Cartridges for Clean-up of Food Prior to HPLC Carbohydrate Analysis

The large variety of food samples routinely submitted for HPLC carbohydrate analysis present matrices which may contain high levels of starches, lipids and/or proteins. These components represent excipients in the sample extract which will either create interfering peaks in the chromatographic analysis or contribute to contamination of the analytical column packing material. Even with the use of an in-line guard column, shortened column life will occur. This creates the need for better sample preparation techniques for "cleaner" sample extracts.

Traditionally, solid phase extraction (SPE) devices such as the Sep-Pak Cartridge have been used to generate sample extracts which contain less mass of matrix excipients.

Sep-Pak Classic and Plus cartridge configurations contain about 0.4 g of sorbent. When using C<sub>18</sub> functionality, this mass of sorbent provides capacity for about 5 mg of retained mass. This dictates that large dilutions would have to be made in order to effectively cleanup a sample using these cartridge configurations. A 1/100(w/v) dilution of a 12% protein content food would, for instance, contain 1.2 mg of protein per mL of extract. Hence, a 4 mL volume of this extract (even assuming it contains no lipids or starches) is all that one can "clean up" with the Classic or Plus Sep-Pak cartridge configuration. This large dilution may be acceptable

for sweetened food products, but analyzing natural levels of carbohydrates in foods requires larger mass extractions (10/100, w/v). This high concentration sample extract presents a problem to traditional Sep-Pak Cartridges. The extracts contain a large mass of excipient material which will quickly overload the SPE device and result in a sample extract which is less than desirable for injection into the HPLC system.

Recently, Waters introduced a new family of large volume, large capacity Sep Pak cartridge configurations. The syringe barrel design of these cartridges is not only easy-to-use, but also lets you choose the amount of sorbent needed for a given sample. Cartridges with up to 10 grams of sorbent mass are now available. One useful configuration consists of a 12 mL syringe barrel containing 2 g of  $C_{18}$  sorbent allowing for a 4/100 (w/v) extraction where a 1/100 (w/v) had previously been done. This provides the ability to inject 4X the amount of sugars into the HPLC for better capabilities of low level analysis while preserving the separation characteristics of the analytical column. See page 30 in the Ordering information section.