

The



# Analytical Digest

WEST COAST ANALYTICAL SERVICE INC

THE QUARTERLY NEWSLETTER ON PROFESSIONAL ANALYTICAL CHEMISTRY

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## Calendar

Oct 27-29, 2004  
Water Quality Conference  
Double Tree Hotel  
Ontario, California

Nov 7-11, 2004  
American Association of  
Pharmaceutical Scientists  
(AAPS) Show  
Baltimore, Maryland

## Quick Quotes

"Before I got married I had six theories about bringing up children; now I have six children, and no theories."

- John Wilmot,  
Earl of Rochester

"Growth for the sake of growth is the ideology of the cancer cell."

- Edward Abbey

## CHN Analysis

WCAS now offers CHN elemental analysis (carbon, hydrogen, and nitrogen) in organic materials using an Exeter Analytical CE440. Samples are combusted at high temperatures in a stream of oxygen, and the instrument measures the products of combustion for carbon, hydrogen, and nitrogen in a single analysis. This instrument offers unsurpassed precision and accuracy. The absolute error in the measurement is 0.3% by weight. Detection limits are generally 0.01-0.1%. Sample sizes are typically 1-10 mg weighed accurately on our new microbalance—a Mettler MX5.

### OTHER ELEMENTS

This system can also be used for sulfur analysis, but we do not currently offer this service. Instead sulfur is measured separately using oxygen bomb combustion followed by ion chromatography. This approach is appropriate for chlorine (Cl), bromine (Br), sulfur (S), and phosphorus (P) in the same analysis. Trace metals are analyzed by ICPMS, ICPOES, GFAA, and XRF. Please contact our client services department should you be needing elemental analysis. ■

## Is Perchlorate Everywhere?

Our scope of perchlorate testing is continually expanding. We have incorporated an internal standard into our methodology, which greatly improves precision and accuracy in the analysis of difficult samples. We recently tested several dairy products, including whole and nonfat milk, soy milk, and yogurt. All have contained measurable perchlorate, ranging from 2-20 ppb. In an effort to identify the source of the perchlorate, one dairy farm sent three samples of alfalfa, which ranged from 55 to a whopping 1900 ppb. This is far in excess of what we have typically seen in produce samples, and seems to confirm the alfalfa as the source of perchlorate in milk.

We have also been investigating other foods for potential perchlorate, and it seems to be everywhere. As we complete more of this work, we will continue to keep you informed. If you have samples you would like tested for perchlorate, please call Eric Lindsay at extension 300 or Louis Albanese at extension 303. ■

## Trace Metals in Fish

WCAS is pleased to announce that we participated in an international, interlaboratory study on arsenic (As), lead (Pb), mercury (Hg), and selenium (Se) in fish. The study was conducted by the Institute for Reference Materials and Measurements

(Belgium). The study consisted of a single sample of freeze dried tuna fish (IMEP-20). The WCAS results were in excellent agreement with the study means. The study details are available from IMEP. ■

### IMEP-20 RESULTS

	Certified Value	WCAS
Arsenic	4.93	5.10
Lead	0.498	0.490
Mercury	4.32	3.50
Selenium	6.38	6.50

## Lab Notebook

We will be attending the **AAPS Annual Meeting and Exposition** in Baltimore, Maryland from November 7-11, 2004. Our booth will be at 360. If you are attending please come by and visit with **Eric Lindsay, Ramona Northington, and Dr. Jack Northington**. We have made many improvements over the last year and are excited to tell you about them.

The LC group has added a **new HPLC** to handle both normal phase and reverse phase applications. The instrument is a Dionex "SUMMIT" system with all stainless steel tubing, quaternary pump, multi-wavelength UV/Vis detector, column heating accessory, and runs on 'Chromleon' software. Initial plans are to set up some special normal phase applications for pharmaceutical products.

The **East Valley Water District** is putting on a Water Quality Conference from October 27-29 in Ontario, California. The conference will focus on perchlorate, disinfection by-products, arsenic, and pharmaceuticals which is a perfect conference for us as we have experience testing for all of these! We will have a booth there so would be more than happy to talk to any of you about testing for the above.

## Elemental Speciation

The toxicity of various elements like Arsenic (As), mercury (Hg), and chromium (Cr) depend greatly on the valence state or chemical form. For many years we have provided specific tests for Cr(VI) and various forms of arsenic and other elements.

Recently we purchased a new Agilent 7500ce ICPMS with both LC and GC interfaces in hopes of expanding our capabilities in this area. Earlier we published results for determining methylmercury (MeHg<sup>+</sup>) using LC-ICPMS. We revisited methylmercury with our new Agilent LC-ICPMS and developed more robust chromatographic and extraction conditions with excellent results. We also found that similar LC conditions could be used for determining ethylmercury, phenylmercury, and thimerosal.

Future plans for the new Agilent system include developing a more sensitive organotin analysis with a larger number of target analytes than just the butyltin series. GC-ICPMS has been reported to give much better sensitivity than the current GC-FPD method. We will also be working on selenium (Se) speciation, expanding that analysis to include cyanoselenate in addition to selenite and selenate. Cyanoselenate is currently of interest in refinery wastewater. ■



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## The **WCAS** Analytical Digest

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## Carbonate by IC

Carbonate and bicarbonate can be determined in natural water samples using pH and alkalinity. The balance between carbonate and bicarbonate is pH dependent. But with small samples (mg) or more complex samples (peptides), one can determine carbonate directly using ion exclusion chromatography. The chromatogram of a peptide sample spiked with carbonate below was conducted on a

Dionex system. The ICE-AS6 column uses 1.6 mM heptafluorobutyric acid as an eluent with suppressed conductivity detection.

In ion exclusion chromatography, the anions of strong acids (like Cl<sup>-</sup>) are not retained, eluting in the void volume. Anions of weak acids are retained. This technique can also be used for determining borate. ■

Chromatogram of a peptide sample spiked with carbonate

