

The



Analytical Digest

WEST COAST ANALYTICAL SERVICE INC
THE QUARTERLY NEWSLETTER ON PROFESSIONAL ANALYTICAL CHEMISTRY

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Feb 27-Mar 4
Pittconn 2005
Orlando, FL

Mar 13-17
ACS National Meeting
San Diego, CA

Quick Quotes

A life spent
making mistakes
is not only more honorable
but more useful
than a life spent doing
nothing.

- George Bernard Shaw

Funny how a dollar
can look so big
when you take it
to church,
and so small
when you take it
to the store.

- Frank Clark

Tween 80 by LCMS

Tween 80 (also known as Polysorbate 80) is used in the manufacture of protein solution formulations to help solubilize and stabilize the protein. It is one of a series of materials (including Tween 20, 40 and 60) which are fatty acid esters of sorbitan polyethoxylates. The various Tweens differ in the type of fatty acid present; Tween 80 is an oleate.

Tween 80 is added into the formulation at relatively high concentrations (~0.1%), and then removed later in the manufacturing process. WCAS has developed an LC-MS method to determine the residual Tween 80 concentration down to approximately one part per million.

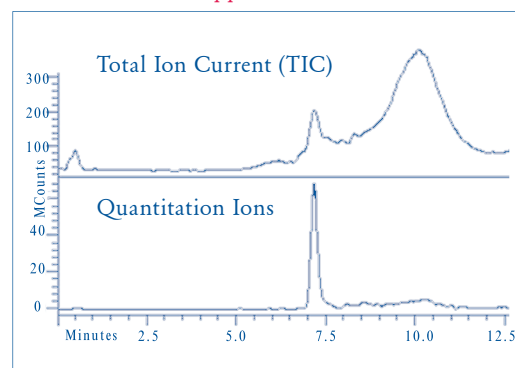
The LC separation yields a series of overlapping peaks, since Tween 80 is a mixture of isomers and congeners. Ionization is accomplished using electrospray, which gives multiply charged ions. By selecting a group of ions which correspond to a narrow band of congeners, a single chromatographic peak is obtained from the complicated Total Ion Current chromatogram (see figure at the right).

For samples with high levels of Tween 80 (~0.05% or higher) or low salt concentrations, the samples are diluted into water and analyzed directly. For lower concentration samples, solid-phase extraction is used to separate the Tween 80 from the protein and buffer salts, which

can adversely affect electrospray ionization. In either case, standards are prepared in the same manner as the samples, which accounts for any potential losses due to the preparation. This enables determination of Tween 80 from samples which originally contained high concentrations (>100 mM) of buffer salts.

We have seen some evidence that Tween 80 from different sources can yield somewhat varying results. For that reason, it is recommended that a sample of the Tween actually used in the manufacturing process be supplied with any samples submitted for analysis. The method currently in place is applicable only to Tween 80 in the absence of other Tweens; we are investigating approaches for samples which may contain more than one. ■

Chromatogram of a Protein Solution
~17 ppm Tween-80



MW by Electrospray MS

Electrospray mass spectrometry is a "soft" ionization technique which can be used to determine the molecular weight of many types of organic compounds. The main requirement is that the molecule can form an ion in solution under electrospray conditions. We have used this technique to determine molecular weights of ¹⁴C labeled compounds, demonstrating that they have been successfully

labeled. Analysis of compounds which do not ionize easily may still be accomplished with the addition of a salt (usually ammonium formate) as a source of ions. In electrospray, these ions will frequently transfer a charge to the neutral analyte molecule, making it detectable as a protonated molecular ion (positively charged) or deprotonated molecular ion (negatively charged). ■

Lab Notebook

We are pleased to announce an update in our FTIR equipment. A **Thermo-Nicolet Avatar 370** equipped with a total reflectance cell has just been installed. This will allow us to analyze smaller samples and surfaces.

Mike Shelton will be presenting a poster at Pittcon in Orlando, Florida in late February. The poster is titled "Perchlorate in Drinking Water and Foods by LC-MS/MS"

and summarizes the results of our analysis of scores of samples from around the country. These include drinking water samples from California, Arizona, Nebraska, Oklahoma, Illinois, Canada, and others, as well as foods ranging from lettuce to milk to baby food.

This represents several months of methods development and analysis to cover the variety of matrices involved. For those of you not able to attend, the basic conclusion

is that perchlorate seems to be everywhere. A copy of the poster will be available on our website at www.wcas.com sometime in early March, following the meeting.

As always please feel free to contact our **Client Services** department with any questions or if you would like to submit samples at **562.948.2225**.

Our next newsletter will be dedicated to **Inorganic analysis** and information.

Selenocyanate by LC-ICPMS

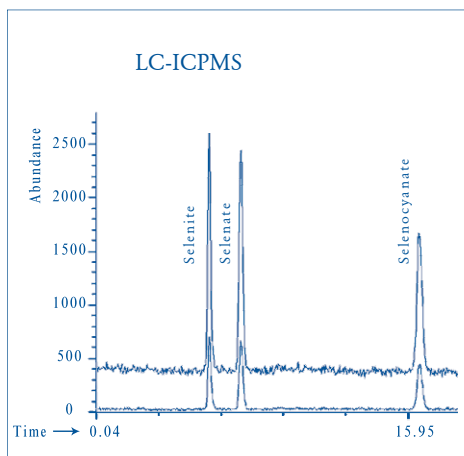
Selenocyanate is a concern in wastewaters from petroleum refineries. Much of the selenium has been characterized as selenocyanate ion (SeCN^-). With our new Agilent 7500ce and HPLC interface, we have implemented this analysis following the work of Wallschlager and Roehl, JAAS, 2001, 16, 922-925.

While selenite (Se^{+4}) and selenate (Se^{+6}) separate by ion chromatography under mild buffer conditions, selenocyanate requires a much stronger eluent. The stronger eluent requires a chemical suppressor as part of the LC-ICPMS interface.

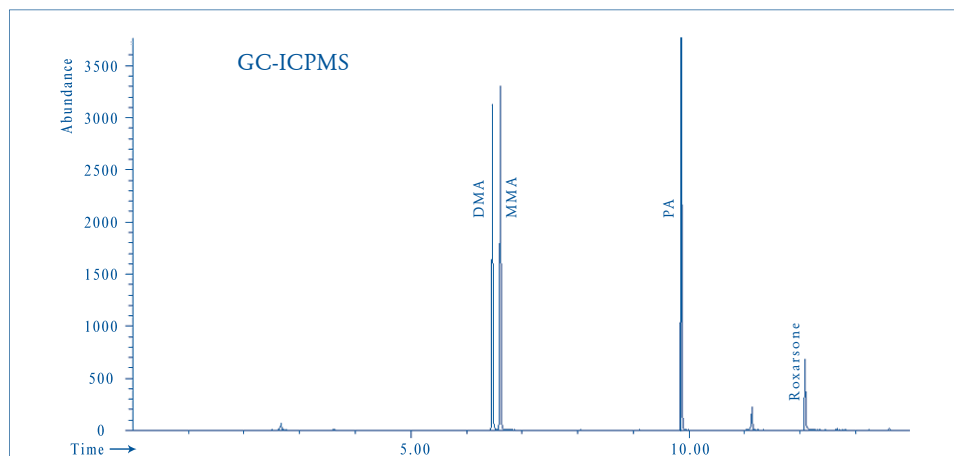
The chromatogram below is a 10 $\mu\text{g/L}$ standard showing the separation of the three species of concern. Detection limits are approximately 1 $\mu\text{g/L}$. Two characteristic isotopes of selenium (77 and 78) are monitored in the chromatogram.

If you have any questions regarding selenium testing or if you would like to submit samples, please contact our Client Services department at 562.948.2225. ■

10 $\mu\text{g/L}$ Standard



100 $\mu\text{g/L}$ Arsenic Speciation Standard



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Arsenic by GC-ICPMS

Various arsenic species react with 1,3-propanedithiol (PDT) to form relatively volatile cyclic dithiaarsenolines. Adapting the method of Roerdink and Aldstadt (J. Chrom. A, 1057, 2004, 177-183) we have developed a GC-ICPMS procedure that can be applied to monomethylarsonic acid (MMA), dimethylarsenic acid (DMA), phenylarsonic acid (PA), and roxarsone. An aqueous sample or extract is simply acidified with hydrochloric acid, reacted with a small amount of PDT, and the reaction products

are then extracted. The chromatogram below shows the separation and quantitation of these compounds. The ICPMS monitors mass 75 characteristic of arsenic. Detection limits are ~1-10 $\mu\text{g/L}$ using a 10 mL sample. One of the advantages of this method is the much greater resolving power provided by capillary gas chromatography (GC) over that possible with liquid chromatography (HPLC). Peaks from LC or IC are generally 20-30 seconds wide, while these capillary GC peaks are ~2 seconds wide. ■