The WCAS Analytical Digest

WEST COAST ANALYTICAL SERVICE INC

THE QUARTERLY NEWSLETTER ON PROFESSIONAL ANALYTICAL CHEMISTRY

ICPMS in 1988

Contents

Calendar

June 5-8
AAPS Biotech Conference
San Francisco, CA

Nov 6-10
AAPS Meeting and Expo
Nashville, TN

Quick Quotes

People will buy anything that's one to a customer.
- Sinclair Lewis

Progress is a continuing effort to make the things we eat, drink and wear as good as they used to be.
- Bill Vaughan

20 Years of ICPMS

In our 20 years experience, we've come a long way with ICPMS, and have many fond memories. We would like to take this opportunity to share just some of the highlights with you.

1985–In the beginning...

Purchasing a VG ICPMS for metals analysis was a gamble—a gamble that proved worthwhile. ICPMS continues to be the most sensitive technique for measuring metals in solution, as well as one of the fastest techniques for multi-element measurements.

1988

Our second VG instrument was purchased. We presented a poster on our software for off-line data reduction at an ICPMS conference in England. The software allowed us to calculate sample results with detailed QC and flexible, client specific results. The software has gotten more powerful since then, and we still use it today.

This was also the year that US Borax approached us about developing methods for analysis of trace boron in a variety of biological specimens, food, and other materials. This was aimed at supporting their research on the toxicology and nutritional essentiality of boron. Over the next few years, the technique was applied to thousands of samples from universities where US Borax was sponsoring research.

Several presentations were made at scientific meetings and papers published. Shortly after this, a similar study was performed for iodine in biological samples for another sponsor. Today the analysis of biological samples for a variety of elements has become routine.

US EPA invited us to participate, along with other ICPMS pioneers, in developing what eventually became Method 6020. Work was performed in validating both EPA Methods 6020 and 200.8.

1990 to 1992

We purchased our third VG instrument. Around this time we developed a hydride generation system for measuring ultra-trace levels of arsenic in water. Detection limits were less than 0.01 µg/L. The chemistry was also adapted to distinguish between inorganic arsenite (As^{+3}) and arsenate (As^{+5}) at this low level. And in 1992, our fourth VG instrument was uncrated and put to work.

1995 to 2000–An era of change...

This became known as our PerkinElmer era. Due to the changes in the industry, it was time to try a different supplier. During this period we purchased 4 PE ELAN 6000's and 6100's. Our last was with Dynamic Reaction Cell (DRC). In the past, interferences from molecular ions such as ArO^+ and $ArCl^+$ had to be dealt with prior to the ICPMS analysis. This was the single largest drawback of ICPMS. Interferences in the plasma from some acids prevented one from achieving sub-ppb detection limits for some elements such as Fe and As.

Since around 1995, innovative developments in reaction and collision cells have pretty much eliminated most of these problems. Our newest system, an Agilent 7500ce, has nearly zero interferences from typical molecular ions.

A look at the present and future...

Our newest instrument, the Agilent 7500ce, purchased with both HPLC and GC interfaces, has allowed us to develop our speciation capabilities even further. In 2000, Mike Hovanec developed the technique for LC-ICPMS speciation of inorganic and organic forms of arsenic using one of the PE ELAN's. He has applied this methodolgy to a variety of environmental, biological, and nutraceutical samples, while making presentations and publishing papers.

We continue to make progress with our newest system—developing capabilities for speciating selenium, organomercury, tetralkyllead, organotin, organosulfur, and organoarsenic compounds.

It has been fun...

Many people have contributed to this success, too many to thank here. ICPMS is one of our mainstay instrument systems. Our metals analysis group, five ICPMS systems along with one ICP-OES, GFAA, and an XRF, is the largest group in the lab. To think it all got started with a gamble twenty years ago. And on that note we'll end this chronicle to go write the purchase order for our next instrument. ■

Lab Notebook

We will be exhibiting at the **AAPS National Biotechnology Conference** being held on June 5-8 at the San Francisco Marriott. The exhibit will be held on June 6 and 7 in the Golden Gate Ballroom. We look forward to meeting you there and talking about your projects.

Our new **Autopol V Automatic Polarimeter** from Rudolph Research Analytical should be here by the time you read this! It will dramatically improve our capabilities concerning Optical Rotation by USP <781>. It will also allow us to perform method development on new materials. It comes with electronic cooling and heating, and six standard wavelengths. We are very excited about this addition.

We are expanding again! We have leased another 8,000 sq. ft. of additional floorspace and will be moving our sample receiving department and sample storage area into the new space. This will allow us to devote more of our current floorspace to analytical groups, especially our metals testing group.

As always you are welcome to schedule a tour or audit with us at any time! Just give Eric or Louis a call at 562.948.2225 (extensions 300 and 303 respectively).

Technical Articles

Here are just some of the many technical articles related to ICPMS and inorganics analysis that can be found on our website at wcas.com:

- GFAA vs ICPMS
- Testing for Lead in Supplements using ICPMS and GFAA
- Chromium by ICPMS-DRC
- Analysis of Iodine
- Arsenic Speciation by by IC-ICPMS
- ICP-MS ELAN 6100 DRC
- Selenium Analysis
- ICP-MS Metals Analysis
- XRF SQX Analysis

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Thin Film by XRF

Measuring the thickness of thin films in the range of 0.005 to 10 µm (50 to 100,000 Angstroms) is a difficult challenge. X-ray Fluorescence Spectrometry (XRF) with specialized software is the most widely used technique for measuring the thickness of thin films. This technique is used on a routine basis to measure the thickness of thin films made from most elements in the Periodic Table, whether in pure elemental form or as compounds (such as SiO_2 or TiO_2) on any solid substrate. XRF also has the ability to measure the thickness of each individual thin layer in a multilayer system.

Standards with known film thickness are available for only a few materials, and therefore severely limits the applicability of some techniques for thin film measurement. XRF however can use bulk standards (i.e. not thin films) which are made from any available high purity material or from standard reference materials with precisely known compositions.

The versatility of XRF to thin film analysis arises from the fact that the theory of thin film measurement by this technique is based upon a very basic equation (1):

$$I = k\,d\,T$$

The equation states that the intensity (I) of fluorescent radiation for a particular element is directly proportional to the film thickness (T) and density (d). The constant (k) is determined from the analysis of bulk standards of known composition based on the Fundamental Parameters X-ray theory. Using densities from reference tables allows us to determine T. Conversely, if the thickness is known by some other technique, the density of the layer can be determined. The technique is discussed in detail by Chung, et al (2).

Please feel free to call our client services depament at 562.948.2225 should you be needing thin film testing or other XRF analysis. ■

1. Ron Jenkins, W. Gould and Dale Gedcke; Quantitative X-ray Spectrometry, Second Edition, Practical Spectroscopy Series, Volume 20, 1995, page 353.
2. F. H. Chung, A. J. Lentz, and R. W. Scott, X-Ray Spectrometry, 3: 172 (1974).