Agilent ICP-MS Journal

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Celebrating 50 Issues of the ICP-MS Journal

The Measure of Confidence

Agilent Technologies

Chemical Characterization of Alligator and Dwarf Crocodile Skin by LA-ICP-MS

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Introduction

The Natural History Museum in South Kensington, London is a world-renowned visitor attraction and natural history research institute. One aspect of the museum's work is to bring its vast collection into contact with the public, especially to excite children about science. Much of the museum's collection has been acquired since Victorian times, and preservation methods that were used on specimens in the past may now be deemed hazardous or unsafe. It is therefore essential that specimens be properly vetted prior to use in public interactive displays.

The Imaging and Analysis Centre (IAC) consists of experts who run both imaging and analytical instruments which are utilized by the entire museum, external researchers and for external contract work. Along with solution ICP-AES and ICP-MS the analytical chemistry section houses a dedicated laser ablation (LA-)ICP-MS.

The learning department wished to use two crocodilian specimens (an alligator (pictured Figure 1 and 2) and a dwarf crocodile (pictured Figure 2)) within the interactive exhibits for children. The specimens were from within the museum's collections and were preserved after the animal's death. However, little was known about the preservation techniques used and of particular concern was that an arsenical soap may have been used to preserve the skin. The IAC was asked to provide a general chemical characterization of the skin of both specimens and to assess the concentrations of elements such as arsenic, which may be harmful if transferred to a visitor's skin on handling the specimen. LA-ICP-MS was used as it offers a quick, relatively non-destructive technique to chemically characterize the skin of the specimens.

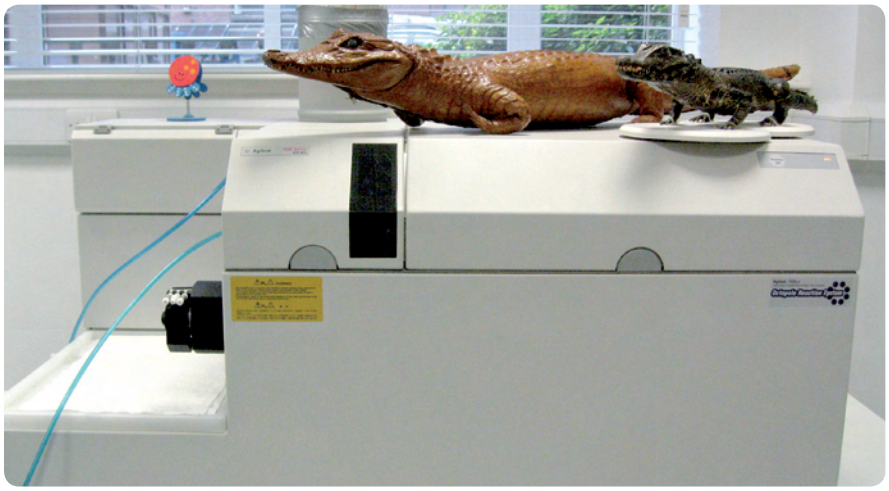


Figure 2. Alligator and dwarf crocodile with Agilent 7500cs ICP-MS

Experimental

Sampling

Fragments of skin were carefully removed from less visible regions on the specimens, using a scalpel and tweezers (as shown in Figure 1). Two fragments were taken from each specimen and laid either face up or face down on double sided tape. The tape was placed on a glass slide and mounted into the sample holder, prior to insertion into the laser ablation cell.

Figure 1. Emma Williams sampling skin from below the jaw on the alligator specimen.

Instrumentation

An Agilent 7500cs ICP-MS (Figure 2) coupled with a New Wave UP193FX laser ablation system were used for the analysis of both the dwarf crocodile and alligator skins. The operating conditions of the 7500cs are detailed in Table 1 and the instrument was tuned to give an oxide ratio <1% and a doubly charged ion ratio <0.1%. Ablation was performed using He as the laser carrier gas. The laser properties were adjusted to be less aggressive than those used for routine mineral ablation due to the organic composition of the skin. A repetition rate of 10 Hz, and fluency of 1.5 Jcm^{-2} was used during ablation with a spot size of 80 um.

ICP-MS operating parameters	
RF power	1500 W
Carrier gas flow	1.1 L/min
Option gas flow	44.5%
Data acquisition	Time resolved mode Peak jumping: 10 ms per peak

Table 1. Operating conditions for Agilent 7500cs during analysis of crocodilian specimens.

Results

Quantitative analysis was not possible due to the unknown ablation properties of the skin, the lack of a suitable reference material and no known elemental concentration that could be used as an internal standard. However, qualitative measurements were possible by monitoring the change in signal for each element as the samples were ablated. The relative values of elements such as As, Cr, Hg, Pb, Sn, V were monitored using time resolved analysis (TRA), as illustrated in Figure 3.

Time resolved data for the dwarf crocodile skin (Figure 3A) showed no traces of As but some Pb content. However, the Pb content is much lower than the ambient surface contamination. The dwarf crocodile has a very uniform skin composition. No chemical layering of the skin or surface coatings (such as varnish) were identified.

The TRA plot for the alligator skin (Figure 3B) showed distinct layers of chemically different compositions. One layer has an As content significantly above the background level. This was seen on the depth profiles of both the face-up and face-down skin samples. We were able to identify that the As containing layer is not present on the surface of the specimen ('varnish' on Figure 3B), but may be exposed in areas where the top coatings had worn off.

Conclusions

LA-ICP-MS using an Agilent 7500cs allowed for non-destructive chemical characterization of the skin of two specimens from the Natural History Museum's collection. Time resolved analyses of several elements revealed differences in the chemical composition of the layers of both specimens' skin. Analysis of the alligator skin revealed arsenic within one layer of the skin, but the absolute concentration was not quantified.

The results allowed the learning team to make an informed decision and to take appropriate steps to ensure that the specimens on interactive display could not cause harm to visitors.

The dwarf crocodile was successfully used in an interactive display to compare modern reptiles to the Spinosaurus dinosaur as part of the BBC Planet Dinosaur 'build a dinosaur' exhibit at the museum.

Learn more at:
www.nhm.ac.uk/research-curation/science-facilities/analytical-imaging

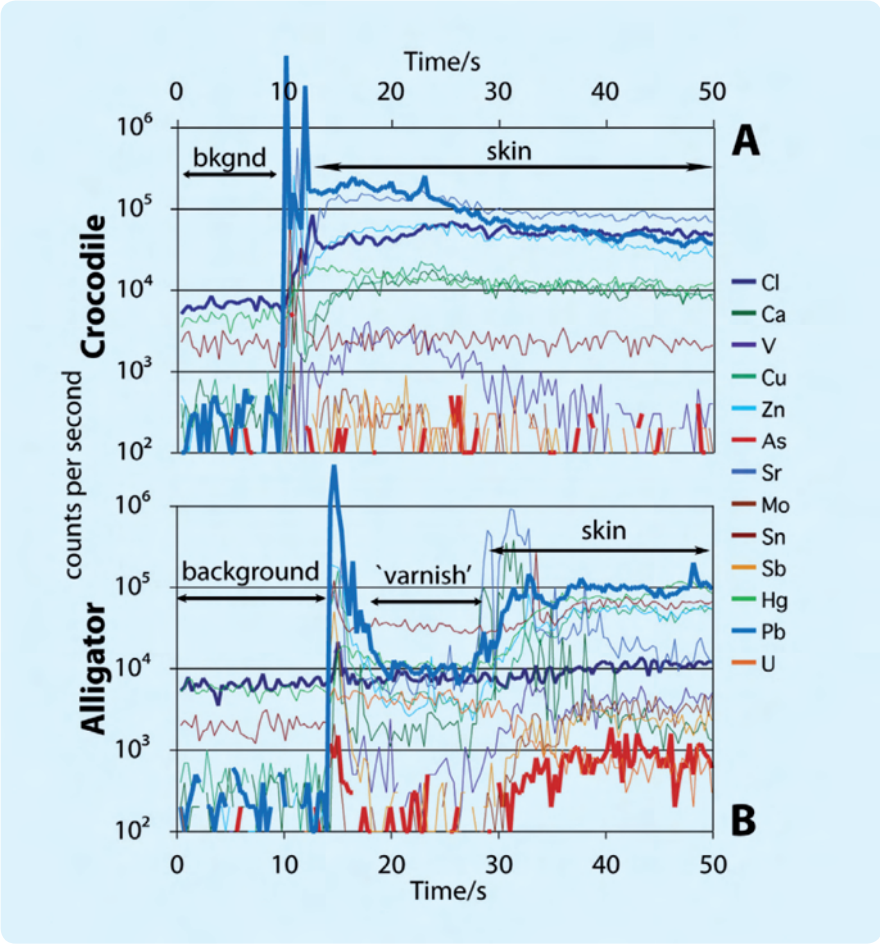


Figure 3. Intensity vs. time signals for elements of interest As (red), Cl (purple) and Pb (blue) in bold.
(A) Crocodile skin – no layering; relatively homogeneous skin composition.
(B) Alligator skin has distinct layers with different Pb levels. As present in deeper layer and behaves independently of Cl.

Four New Atomic Spectroscopy Food Safety Application Notes Now Available

Steven Wilbur
Senior Applications Chemist,
Agilent Technologies

Increasingly, the quality of the foods and beverages we consume is brought into question in the scientific literature, on the internet and on television and radio. New discoveries of toxic contaminants in unexpected places are regular events. In part, these discoveries are the result of vastly improved surveillance, monitoring and regulatory programs which are intended to prevent human exposure to unsafe foods. Improved analytical capability also plays an important role – a role which Agilent Technologies takes very seriously.

Three of the new food safety application notes described herein are examples of ways in which significant advancements in ICP-MS technology have been applied to the determination of trace elements and elemental species in a range of food and beverage products. The fourth note describes the analysis of Chinese herbal medicines using microwave plasma-atomic emission spectrometry.

1. Enhancing the productivity of food sample analysis with the Agilent 7700x ICP-MS, 5991-0107EN
2. Benefits of HPLC-ICP-MS coupling for mercury speciation in food, 5991-0066EN
3. Trace elemental analysis of distilled alcoholic beverages using the Agilent 7700x ICP-MS with octopole collision/reaction cell, 5990-9971EN
4. Analysis of Chinese herbal medicines by microwave plasma-atomic emission spectrometry (MP-AES), 5990-9791EN

These new notes can be downloaded at **www.agilent.com/chem/atomic**

Analysis of foods for trace element content has always been a challenging problem. Food matrices are complex and variable, and the toxicologically relevant concentrations of many trace elements can be quite low. Additionally, many elements vary significantly in their toxicity or nutritional value depending on the species. Well known examples include mercury, whose organic forms, particularly methyl mercury are significantly more toxic than the inorganic forms, and arsenic, whose inorganic forms are highly toxic, while many organo-arsenic compounds are thought to be completely non-toxic. Chromium and selenium are other interesting examples. Hexavalent chromium (Cr(VI)) is highly toxic while trivalent chromium (Cr(III)) is an essential micronutrient for humans. Selenium is toxic at high concentrations and exhibits anticancer properties at lower concentrations.


Enhancing the productivity of food sample analysis with the Agilent 7700x ICP-MS

Application note

Foods testing

Authors

Sebastien Sannac, Jean Pierre Lener and Jerome Darrouzes

Agilent Technologies
Paris, France

Introduction

5991-0107EN describes a simple, rapid, microwave digestion procedure followed by ICP-MS analysis which is amenable to a large number of elements in a wide range of food types. The method uses an Agilent 7700x ICP-MS operated in helium collision mode to remove all matrix based interferences regardless of the food matrix. The method was validated via the analysis of multiple certified reference food materials ranging from NIST 1548a (complete diet), to DORM3 (fish tissue), to NIST 8415 (whole egg), with excellent recoveries of all certified values. In addition, since He mode is effective for all polyatomic interferences, it makes secondary isotopes available for many analytes, enabling confirmation of the quantitative results and providing significantly improved confidence in reported values for unknown samples. From a productivity standpoint, the use the ISIS-DS discrete sampling accessory reduced the time taken for the analysis of 16 elements to 1.2 minutes per sample.


Benefits of HPLC-ICP-MS coupling for mercury speciation in food

Application note

Food testing

Authors

Sebastien Sannac, Yu-Hong Chen, Raimund Wahlen, Ed McCurdy

Agilent Technologies
Manchester, UK

Introduction

Agilent Technologies

5991-0066EN addresses two important analytical challenges. The first is the extreme toxicity of organo-mercury, requiring very low limits of detection, and the second is the challenge of simply and rapidly separating the various toxic mercury species without risk of species interconversion. Until recently, Hg speciation has been performed using GC-ICP-MS, mainly because of the better resolution and higher sensitivity. However, GC methods for Hg speciation require derivitization and solvent exchange prior to analysis. In contrast, liquid chromatography based methods can permit direct analysis of the digested sample. This application note demonstrates a simple, sensitive method for the determination of four mercury species in less than 3 minutes using HPLC coupled to ICP-MS as a detector – Figure 1. The highly robust plasma and fast frequency matching RF generator of the Agilent 7700x ICP-MS permit the rapid change from aqueous to 90% methanol (and back) required for this gradient HPLC method. Excellent recovery of certified values for two reference materials (DOLT-4 and BCR 464) was demonstrated with background equivalent concentrations (BEC) of less than 20 ppt for all species.

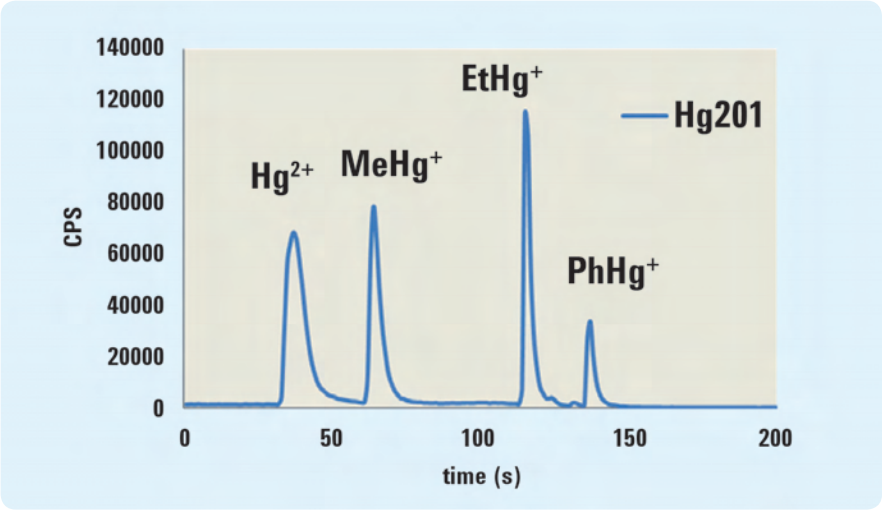
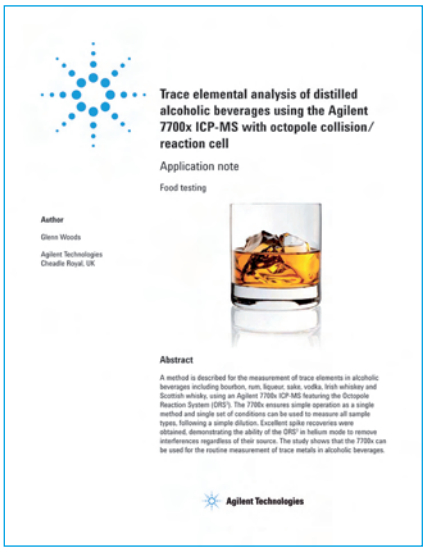


Figure 1. Gradient elution of four mercury species in less than 3 minutes using HPLC-ICP-MS


Analysis of Chinese herbal medicines by microwave plasma-atomic emission spectrometry (MP-AES)

Application note

Food Testing


Trace elemental analysis of distilled alcoholic beverages using the Agilent 7700x ICP-MS with octopole collision/ reaction cell

Application note

Food testing

5990-9971EN deals with the determination of a large number of trace elements in a range of alcoholic beverages, prepared by simple dilution and acidification. The measurement of trace elements in alcoholic beverages is required from a quality control standpoint and also to ensure that the final product complies with any regulatory requirements. Trace element patterns may also be useful in identifying region of origin, which may have a major impact on product value. Elements in beverages can originate from the raw ingredients, such as water or grain, or can be added during processing, for example from fermentation or distillation equipment. An example would be high arsenic concentration that may be leached from distillation vessels manufactured from poor quality copper.

The exceptional robustness of the 7700 ensured that no matrix matching was required and calibration was performed in dilute nitric acid. In order to compensate for sample transport effects and solvent evaporation rates, the alcohol content of the standards was matched to that of the samples by adding ~10% ethanol to all standard solutions. No sample-specific method development or customized cell modes were required, as the use of helium gas mode in the ORS^3 of the 7700x efficiently removed the plasma-based and matrix-based interferences from all analytes. The use of He mode improves detection limits and ensures accurate and reliable results with a simple, consistent set of conditions for all interfered elements, regardless of the sample composition. He mode provides improvements in detection limits of around 4 orders of magnitude for interfered elements compared to no gas mode and is applicable to multielement analysis in a wide range of sample types.

5990-9791EN discusses the determination of a suite of elements, including heavy metals, in three typical herbal medicines prepared by microwave digestion for analysis by Microwave Plasma-Atomic Emission Spectrometry (MP-AES).

Traditional Chinese herbal medicines (TCMs) and their related products have been widely used in China for centuries; however there is now greater focus on the safety issues related to TCMs in clinical use. The analysis of both toxic and beneficial heavy metals is crucial in quality control of TCMs, and regulations have been implemented to control their levels. In order to enhance TCM safety and management, the Chinese Pharmacopoeia (2010 edition) was created in July 2010 and this is now the standard for Chinese medicinal crops, Chinese patent drugs, and Chinese medicinal herb plant material and extracts.

The results obtained using MP-AES were in good agreement with those obtained using conventional atomic spectroscopy techniques. The recoveries of the MP-AES method were between 90 and 110% and the method detection limits for most elements were less than 3 ppb in the solid sample. The recovery and precision were also excellent demonstrating the ability of the Agilent 4100 MP-AES to meet the needs of a very demanding industry.

The method was confirmed by analyzing a leaves of aspen (GBW07604) Certified Reference Material from National Standard Substances which is often used for quality control of TCM analysis. The measured values were in good agreement with the certified values.

MP-AES provides an ideal, low-cost solution for multi-element analysis of TCMs, with excellent long-term stability, reduced running costs, and improved lab safety due to the absence of flammable gases. It is suited to food applications that require the analysis of a medium number of elements per sample with detection limits typically in the ppb range.

Trade-in Your Used Agilent Platinum ICP-MS Cones and Save Money!

Eric Vanclay
Spectroscopy Supplies Product Manager,
Agilent Technologies

The Agilent 7700 Series ICP-MS has set the benchmark for quadrupole ICP-MS performance, interference removal and size. Even the best ICP-MS system needs to be optimized for maximum performance.

Performance optimization includes checking the condition of the sampler and skimmer cones. The cones are designed to provide the ideal combination of matrix tolerance and high sensitivity. The size and shape of the cones is critical and influences many aspects of instrument performance.

Periodic cleaning of the cones is essential to remove any build-up of foreign matter and maintain peak performance. Even with repeated cleaning, the cones will eventually become unusable, after extended use.

Figure 1: Used platinum cones can now be returned for a trade-in credit and recycling.

Many users keep their used cones in a drawer in their laboratory just in case they might use them again in the future. This rarely happens. Now you can recycle your used platinum cones and save money!

To reduce environmental impact, conserve highly valuable platinum supplies and deliver cost savings to your laboratory, Agilent has now established a trade-in program for used platinum cones.

Using this program, you receive a trade-in credit against return of used platinum cones when purchasing like-for-like replacements. Agilent platinum sampling and skimmer cones for the 8800/7700/7500 Series ICP-MS qualify for the trade-in credit. The value of the credit is based on the market value for platinum, and will be adjusted as platinum prices fluctuate.

This program reduces your net purchase price for replacement platinum cones and allows Agilent to initiate recovery and recycling of the valuable platinum content in the cones.

How the Trade-in Program Works

When ordering new platinum cones, your order processor will ask if you have any used platinum cones to return. If so, a "return order" will be created for the cone(s) to be returned. You will be sent return instructions together with an Environmental Health and Safety (EHS) form and cleaning instructions. After cleaning, the cones should be sent to the address indicated in the documentation together with the completed EHS form. Once received at Agilent, we will confirm the type of cone(s) and the quantity received. A credit will then be applied to your account. The used cones must be received by Agilent within 60 days of the initiation of the order. A maximum of three cones may be traded in on the purchase of a new cone. Please note that the cone(s) that you are trading-in needs to be the same type as the cone(s) being purchased.

Agilent ICP-MS Supplies Maintain Peak Performance

Agilent also maintains a full range of supplies for the 8800/7700/7500 Series ICP-MS from our proprietary torch, through our sampler and skimmer cones to our unique electron multiplier detector. See the full range of consumables in the Agilent Atomic Spectroscopy Supplies Catalog, pub number: 5990-8767EN; Japanese version: 5990-8767JAJP.

With Agilent ICP-MS supplies and accessories, the difference is in the details. All of our products have been engineered or selected by our instrument design teams, manufactured to our tight tolerances and stringent specifications, and rigorously tested under a variety of conditions. This painstaking approach – registered to ISO 9001 – ensures that every part will perform at optimal levels to help you minimize costly downtime. This ensures that you'll always get the best performance from your instrument and the best results for your clients. Only Agilent supplies are designed to work seamlessly with the 8800/7700/7500 Series ICP-MS instruments.

Let Agilent's Platinum Cone Trade-in Credit Program Work for You!

To learn more about how the trade-in credit on used Agilent platinum cones for the 8800/7700/7500 Series ICP-MS can help you save money, be environmentally responsible and improve the reliability of your results, go to **www.agilent.com/chem/PtCone**

Important note:
At the time of publication, the Pt Cone Trade-In Program is available only in North America and Western Europe. We are actively working to bring this program to additional geographies. For further information, go to **www.agilent.com/chem/PtCone**

Agilent Atomic Spectroscopy eSeminar Series

Learn useful tips for troubleshooting and maintaining your Agilent AA, ICP-OES, or ICP-MS to improve uptime and performance. To access the on-demand webinars, go to: **www.agilent.com/chem/AtomicSpecEseminars**

- ICP-MS Troubleshooting & Maintenance
- Elemental Speciation Made Easy and Robust with Separations Systems Interfaced to an Agilent ICP-MS
- ICP-OES Troubleshooting & Maintenance
- AA Troubleshooting & Maintenance

Agilent Passes 1000th 7700 Series Landmark in Record Time

At the end of October 2011, Agilent's Tokyo Analytical Division (TAD) proudly shipped the 1000th Agilent 7700 Series ICP-MS to the ADEKA Corporation located in Tokyo, Japan.

The company has two product groups: Chemicals and Foods. The Chemical Group research, develop and produce a line of products such as high purity materials, including electronic substrate etching products, for the semiconductor industry. ICP-MS is a key analytical tool used by the company's research center during the research and development of new products and for Quality Assurance testing of their final products. The newly acquired 7700s joins an existing Agilent 7500 to manage an increasing sample load.

The ICP-MS team in ADEKA are committed to keeping up-to-date with their ICP-MS knowledge and regularly organize study meetings to share best practice. They believe it is critical to understand the ICP-MS technology well in order to get the best performance from their instruments.

To commemorate the shipment of the 1000th 7700, the instrument was engraved with a special serial number "1000" and adorned with colorful ribbons prior to shipment (picture).

Pictured from left to right: Agilent's Mitsuki Goto with T. Watanabe and A. Hashizume, ADEKA R&D Division Chemical Analysis Group Manager and Group Chief respectively.

Agilent's ICP-MS Pedigree

The 7700 Series is the third generation of Agilent's successful ICP-MS product line. In 1994, Agilent launched the 4500 Series, the world's first computer-controlled, benchtop ICP-MS. The 4500 was replaced in 2000 by the 7500 Series, which revolutionized the analysis of complex samples with the introduction of the unique Octopole Reaction System (ORS) collision/reaction cell, operating in helium mode.

The 7700 Series ICP-MS was released in 2009 and has quickly become the best selling ICP-MS system today. It provides unparalleled levels of matrix tolerance, further enhanced interference removal, and a range of ease-of-use and productivity tools.

The unprecedented success of the 7700 Series is a testament to the quality, reliability and performance of Agilent ICP-MS systems over the past 15 years.

For more information about the ADEKA Corporation visit **www.adeka.co.jp/en/index.html**

Agilent Opens A New Spectroscopy Demo Lab in Waldbronn Facility

The new Spectroscopy Demonstration Laboratory that was opened in Waldbronn, Germany in November 2011 is proving to be extremely popular with our customers and potential customers for evaluation of their samples, demonstrations, meetings and workshops. It is also continually updated with the latest technology. For example a new 8800 ICP-QQQ will be installed in June.

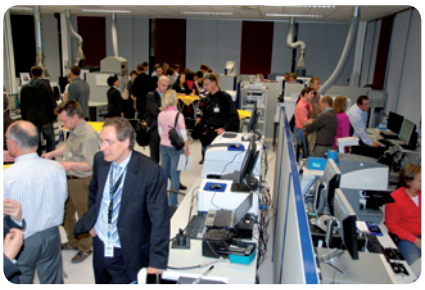
The Grand Opening

Almost 100 users and decision makers from industry academia and research institutions came together to celebrate the opening of the facility. The program began with seminars on atomic and molecular spectroscopy, focusing on Agilent's new products such as the 4100 MP-AES and the 630 FTIR. In parallel, the German ICP-MS user-meeting took place with Agilent contributors and customers delivering high-level presentations on their day-to-day work and research with their Agilent ICP-MS.

Agilent Germany's General Manager Fred Strohmeier cutting the ribbon for the new Spectroscopy demo lab in Waldbronn.

The official lab opening started with a short welcome by Agilent's Fred Strohmeier, Country General Manager for Germany, followed by an update on customer education in the area of spectroscopy.

"The opening of this spectroscopy demo lab is a milestone on our way to serving our customers perfectly", said Tobias Gysin, EMEAI Business Manager Atomic Spectroscopy. "Our customers can find the complete range of spectroscopy products in this lab. All products are put in perspective so that we can offer professional support in the evaluation of the right equipment for their needs."



Following the opening ceremony, the guests had the opportunity of participating in further interactive workshops on spectroscopy, GC/MS and LC/MS, or discussing the latest news in an informal atmosphere (picture).

8800 ICP-QQQ Webinar Available On Demand

Recorded Agilent Atomic Webinar: **Solve Challenging Applications Never Possible Before... ICP-QQQ Has Transformed ICP-MS** presented by Agilent's Ed McCurdy.

Learn how the Agilent 8800 ICP-QQQ is redefining application capabilities and research opportunities, and how you can transform your laboratory performance. To access this highly popular webinar, broadcast on Tuesday, April 24, 2012, go to: **www.agilent.com/chem/AtomicSpecWebinars**

Agilent ICP-MS Publications

To view and download the latest ICP-MS literature, go to **www.agilent.com/chem/icpms** and look under **"Literature Library"**.

- **Brochure:** Agilent 8800 Triple Quadrupole ICP-MS, 5991-0079EN
- **Application note:** On-line isotope dilution analysis of selenium using the 8800 Triple Quadrupole ICP-MS with oxygen reaction mode, 5991-0259EN
- **Application note:** The ultratrace determination of iodine 129 using the 8800 Triple Quad ICP-MS with oxygen reaction mode, 5991-0321EN
- **Application note:** Analysis of silicon, phosphorus and sulfur in 20% methanol using the Agilent 8800 Triple Quadrupole ICP-MS, 5991-0320EN
- **Application note:** Enhancing the productivity of food sample analysis with the Agilent 7700x ICP-MS 5991-0107EN

Celebrating 50 Issues of the Agilent ICP-MS Journal

The Agilent ICP-MS Journal is unique among ICP-MS vendor newsletters in having been published continuously since 1998, and has recorded numerous industry, application and product milestones. Highlights include:

- More than **127** application features including **~60** user contributed pieces such as success stories, customer profiles or user articles, cementing the ICP-MS Journal's role as a useful technical reference source.
- Topics have ranged from analyzing the sword of El Cid, the provenance testing of sapphires and high sensitivity semicon apps, to the routine and high throughput analysis of environmental, foods and clinical samples.
- Specific recent applications include:
  - Application of Compound Independent Calibration to Arsenic Speciation (issue **#46**)
  - High Throughput Analysis of Flue Gas Wastewater Samples by ICP-MS (**#46**)
  - Successful Low Level Mercury Analysis using the Agilent 7700 Series ICP-MS (**#45**)
  - Trace Element Analysis of Undiluted Urine by ICP-MS (**#45**)
  - Determination of Brominated Flame Retardants in Plastic by HPLC-ICP-MS (**#43**)
  - Characterization of Nanoparticulates using Field Flow Fractionation-ICP-MS (**#40**)
- More than **30** announcements of significant product developments, all designed to improve the quality of your results.
- Celebration of notable achievements such as recognition of the scientific contributions made by all the recipients of the Agilent-sponsored biennial European Plasma Prize since its inauguration in **2003**, and installation of more than **4000** Agilent ICP-MS in laboratories located in over **80** countries.

We would like your feedback! If you have an opinion on the future direction of the ICP-MS Journal, we would love to hear your views. Please feel free to contact us via e-mail at: **icpms@agilent.com**

You can access the ICP-MS Journal Archive from: **www.agilent.com/chem/icpms**

Conferences. Meetings. Seminars.

- **ASTS Seminar and Tradeshow Tour:** (North America), May and June, **www.agilent.com/chem/ASTS**
- **Nordic Plasma 2012:** June 10-13, Loen, Norway, **www.nordicplasma.com**
- **Spectr'Atom:** June 19-22, Pau, France, **www.spectratom.com**
- **11th European Workshop on Laser Ablation:** June 19-22, Gijón, Spain, **www.ewla.es**
- **Goldschmidt Conference:** June 24-29, Montréal, Canada, **www.vmgoldschmidt.org**
- **Interphex Japan:** June 27-29, Tokyo, **www.interphex.jp/en**
- **NEMC 2012:** August 6-10, Washington DC, **www.nemc.us**
- **Asia Pacific Winter Conference:** Aug 26-29, Jeju, Korea, **http://apwc2012.dankook.ac.kr/**
- **JASIS (formerly JAIMA):** Sept 5-7, Tokyo, Japan, **www.jasis.jp/2012/english/index.html**
- **21st International Symposium of Forensic Sciences ANZFSS:** Sept 23-27, Hobart, Tasmania, **www.anzfss2012.com.au**

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