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New! Agilent High Matrix Introduction Accessory Expands the Capabilities of ICP-MS

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Introduction

Since the introduction of ICP-MS, analysts have had to limit total dissolved solids (TDS) levels to around 0.1 - 0.2%, to minimize signal drift and matrix suppression. Agilent's 7500 Series was designed to routinely handle higher TDS sample matrices (up to 0.3% TDS) than other ICP-MS systems, primarily as a result of an optimized sample introduction system. Features such as the digital drive, high efficiency 27MHz ICP RF generator, wide 2.5mm torch injector and low sample uptake rate give the 7500 Series a more robust plasma, which is better able to decompose these high matrix levels. A low cerium oxide ratio is often used as an indicator of plasma robustness in ICP-MS; the 7500cx typically operates at 1% CeO/Ce, compared to around 2% or 3% CeO/ Ce typically seen on other ICP-MS instruments.

To meet the 0.2% - 0.3% TDS limit of ICP-MS, high matrix samples must be diluted prior to analysis; this dilution step has several disadvantages, including increased risk of sample contamination, possibility of dilution error, and additional sample prep time. To avoid the drawbacks of conventional dilution, Agilent has developed a new technique for the handling of high matrix samples -"aerosol dilution". The new High Matrix Introduction (HMI) accessory enables the 7500 Series to analyze 1% TDS (or higher, depending on the matrix) samples directly eliminating the dilution step and its associated drawbacks. Plasma robustness is significantly improved over conventional ICP-MS (illustrated by the reduced CeO/Ce ratio of 0.2%), which greatly reduces matrix suppression, making the analysis of high matrix samples more reliable



Figure 1. Signal suppression in undiluted NASS-5

and accurate than ever before with ICP-MS.

How Does HMI Work?

The HMI hardware consists of a modified torch mounting and the insertion of a make up gas line between the spray chamber and torch. Nebulizer gas flow is reduced to decrease the efficiency of the nebulizer, and the make up, or "dilution" gas, is increased so the total carrier gas flow into the torch is kept constant. The sample solution aspirated can be 1% or higher TDS, but much less of the aerosol is transported to the plasma, so the plasma is not overloaded by the extra sample matrix. Reduced sample aerosol transport also means that a reduced amount of solvent and water vapor is carried to the plasma. With less water to decompose, the plasma is hotter and therefore more robust. This increased robustness is key to improving the ability of ICP-MS to run very high and variable matrix samples routinely and accurately, and the analytical benefits are demonstrated here.

Figure 1 shows the % recovery of a 10ppb multi-element spike in undiluted NASS-5 seawater standard, compared to a reference 10ppb spike in 1% HNO₃. The 7500cx was optimized at different levels of plasma robustness: 2% CeO/Ce (representing the typical performance of a non-Agilent system), 1% CeO/Ce (typical 7500cx performance), and finally 0.2% CeO/Ce (HMI used). As can be seen, at 2% CeO/Ce, huge signal loss (suppression) in the NASS-5 seawater matrix is observed, with 50% suppression for Sc and Ba, increasing to 85% suppression (only 15% signal

remaining) for Zn (higher ionization potential element). The more robust plasma of the 7500cx handles the matrix much better but still shows 50-65% suppression for all analytes. With the HMI, however, suppression is almost eliminated, the worst case being In (only 15% signal loss). Note that NO internal standard correction was applied to any of the data in Figure 1. This means that, with HMI, undiluted seawater can be run against a 1% HNO₃ calibration. The presence of high levels of easily ionized elements (in this case approx. 3% TDS - mostly NaCl) has little effect on the Zn signal, demonstrating the improved robustness of the plasma with HMI.

A further example of improved robustness with HMI is illustrated in Figure 2, which shows the relative sensitivity of a set of elements across the mass range, in increasing concentrations of a zinc matrix up to 1% (10,000ppm) Zn. Once again, no internal standard correction was used, and less than 20% suppression is observed - even in the 1% Zn matrix. In addition, all elements are closely grouped - there is no mass-dependence to the suppression effect. Minimal suppression and minimal mass-dependant sensitivity change means that the HMI can measure sample matrices ranging from 0% to 1% metal (in this case Zn), using a single aqueous calibration. When internal standardization is used, much less correction is needed, which increases accuracy and makes it easier to choose suitable internal standards. Productivity is increased since there is no need for close matrix matching.

"I believe that the HMI will prove to be a key technology enabler, allowing the application of ICP-MS to higher matrix samples than previously possible." Kenichi Sakata, Senior Scientist, R&D, Agilent Technologies. In practice, the HMI is extremely simple to set up and operate. Sophisticated software algorithms automate tuning and optimization the user simply selects the level of robustness plasma required ("normal 7500cx" or "HMI") and the ChemStation software automatically loads all the appropriate settings. Once the HMI is fitted, the 7500cx can still be operated in standard mode for lower matrix samples, and normal and HMI mode conditions can be switched automatically

the effect of MoO interference on Cd. While the 7500cx already has a low CeO/Ce of 1%, reducing the CeO/Ce to 0.2% with HMI significantly improves the accuracy of Cd measurement in the presence of Mo. With 2ppm Mo present, the observed value for a 1ppb Cd standard is close to 5ppb under normal 7500cx conditions. With HMI, 1ppb Cd can be accurately measured in matrices containing at least 2ppm Mo. In geological applications, a reduced CeO/Ce level improves the quantification of



Figure 2. Signal suppression in Zn matrix up to 10,000ppm (1%)

within a single analysis; there's no need to disconnect any gas lines. Sensitivity is reduced in HMI mode, but since dilution factors are up to 10x lower, net detection limits are the same or slightly improved. More importantly, the influence of the prep reagent blank on reported analyte concentrations is reduced by a factor of 10, so errors and contamination associated with an additional dilution step for ICP-MS are eliminated.



Figure 3. Effect of MoO interference on Cd

The Benefit of Lower Oxide Levels

The CeO/Ce level is almost universally used as an indicator of plasma robustness, but a low oxide level also has an analytical benefit – reduced interferences. Figure 3 shows mid- and high-mass rare earth elements (REE), as the interference from BaO and low mass REE oxides is reduced significantly.

Applications of HMI

The virtual elimination of matrixrelated signal suppression is a very important step forward for ICP-MS: it means that matrix matching to correct for ionization suppression is no longer necessary. Accuracy when measuring variable, unknown matrix samples is greatly improved, and internal standard selection is simplified. The HMI can handle traditional ICP-OES matrices in many cases - often even using the highmatrix sample digests prepared for OES analysis, and thereby eliminating extra sample prep workup. And since the plasma is more robust, sample matrix decomposition is more efficient, which means less routine maintenance on the interface and ion lenses. Applications for HMI include;

- Impurities in high purity metals
- Soil digests (ICP-OES prep)
- Direct analysis of undiluted
- seawater
- RoHS samples
- Landfill leachates (TCLP prep)

- Geological digestions and fusions
- High TDS groundwaters
- Foods samples
- Pharmaceutical matrices
- HPLC-ICP-MS applications which require a high salt buffer

Ordering Information and Compatibility

The HMI is currently available to order with new 7500cx instruments and will shortly be available as an upgrade to existing 7500ce and 7500cx instruments.

New Standard Reference Material for In-situ U-Pb Zircon Dating

S.Elhlou, E.Belousova, W.L.Griffin, N.J. Pearson, S.Y.O'Reilly

GEMOC ARC National Key Centre Department of Earth and Planetary Sciences, Macquarie University, Sydney, Australia

Introduction

U-Pb zircon dating by laser ablation requires a reliable matrix-matched internal standard to correct for elemental and isotopic fractionation. The most commonly used Canadian reference material, known as Harvard 91500 zircon from Ontario, is almost exhausted and GEMOC has searched for a suitable alternative zircon standard for U-Pb dating.

G&J Gem Merchants (Sydney) donated a parcel of large zircon fragments (~1cm across), believed to be from East African pegmatites. There is a range of colors from red to pinkish-red, yellowish-green and brown. The suitability of this GJ zircon as a standard reference material for in-situ U-Pb zircon dating was investigated.

Analytical Techniques

The internal structures of GJ zircons were assessed visually by cathodluminescence (CL) and by BSE imaging using the Electron Microprobe (EMP). The EMP was used to measure major and minor elements to check for homogeneity.

U-Pb zircon dating and trace element analysis were carried out using a New Wave Merchantek UV Nd:YAG 213nm Laser Microprobe



Figure 1. Similar trace element and REE patterns in different-coloured GJ grains

coupled to an Agilent 7500s ICP-MS (LAM-ICP-MS) [1]. The Hf isotopes were measured on a Nu Plasma Multi-Collector ICP-MS with the same 213nm laser system (LAM-MC-ICP-MS).

Element Concentrations (ppm)

MS analyses of 6 GJ grains.

Table 1 summarizes the LAM-ICP-

GJ Red zircon has slightly higher Pb

and Th concentration levels compared

to GJ zircons of other colors. The

red variety was selected as the

standard for U-Pb dating because it

has a suitable U content and higher

Th and Pb than the other GJ

As Figure 1 illustrates, different-

coloured GJ grains show the same

Results

populations.

trace element and REE patterns with slight variations in concentration levels between the different colors.

LAM-ICP-MS U-Pb Zircon Dating for GJ Red

The ²⁰⁶Pb/²³⁸U age 609.7±1.8 Ma (2sd) for GJ Red zircon was determined by LAM-ICP-MS using the 91500 zircon as a calibration standard. This result is within error of the TIMS age of 608.5±0.4 Ma [1]. Figure 2 summarizes the LAM-ICP-MS results.

Four different red GJ grains were dated by LAM-ICPMS (n=43). From grain to grain there were small variations in the U-Pb ages. As a consequence of these slight variations, it is necessary for each individual zircon grain to be dated by TIMS prior to use as a U-Pb zircon standard.

GJ-Colours	Y	Ce	Yb	Lu	Hf*	Pb	Th	U
Red	267 ±24	20 ±1	71 ±5	13 ±1	7800	36 ±2	18 ±3	287 ±13
Yellow	287 ±12	15.3 ±0.5	72 ±4	14 ±1	4630	23 ±1	11 ±1	384 ±42
Brown	304 ±15	15.7 ±0.5	81 ±6	14 ±1	8310	21 ±3	10 ±1	398 ±48
Green-Grey	343 ±43	18 ±0.2	80 ±1	16 ±3	8225	26 ±7	12 ±1	429 ±104
Yellow-Pink	291 ±16	17 ±1	91 ±13	14 ±1	7890	25 ±2	11 ±1	355 ±14
Grey-Yellow	272 ±29	13 ±4	76 ±3	13 ±1	8225	17 ±8	8 ±4	381 ±30

Table 1. LAM-ICP-MS analyses of 6 GJ grains



Figure 2. 206 Pb/ 238 U age for GJ Red zircon was determined by LAM-ICP-MS – 609.7±1.8 Ma (2sd) – using the 91500 zircon as a calibration standard

Hf-Isotope Compositions

The data in this study suggests that the GJ Red zircon is isotopically homogeneous and is consistent within the uncertainty of results from other laboratories. This makes GJ Red a useful alternative Hf isotope standard to the zircon 91500 which has been shown to be isotopically heterogeneous [3].

Conclusions

The GJ-red zircon is chemically and isotopically homogeneous and thus recommended as a suitable zircon standard for U-Pb dating. It is also suitable as a standard for in-situ trace element and Hf isotopic analysis by laser ablation ICP-MS.

The GJ red zircon has been currently

circulated to 22 laboratories around the world and can be supplied with a small service charge from GEMOC (contact: Elena Belousova email: ebelouso@els.mq.edu.au)

References

- 1. Jackson, S.E., Pearson, N.J., Griffin, W.L., Belousova, E.A. The application of LA-ICP-MS to in-situ U-Pb zircon chronology. Chemical Geology 211 (2004) 47-69
- 2. Wiedenbeck, M., Allé, P., Corfu, F., Griffin, W.L., Meier, M., Oberli, F., Von Quadt, A., Roddick, J.C. and Spiegel, W. Three natural zircon standards for U-Th-Pb, Lu-Hf, trace element and REE analyses. Geostandards Newsletter (1995), 19 (1), 1-23.
- 3. Griffin, W.L., Pearson, N.J., Belousova, E.A. and Saeed, A. Comment: Hf-isotope heterogeneity in standard zircon 91500. Chemical Geology (in press, online March 2006)

Sample	N	176Hf /177Hf	2sd	178Hf /177Hf	2sd	176Lu / 177Hf	2sd	176Yb /177Hf	2sd
LAM-MC-ICP-MS GJ Red Zircon	30	0.282015	0.000038	1.46727	0.00007	0.00025	0.00002	0.00742	0.00074
LAM-MC-ICP-MS 91500 zircon	632	0.282307	0.000058	1.46707	0.00069	0.000317	0.000054	0.01145	0.00502
TIMS* 91500 zircon	7	0.282290	0.000014	1.46714	0.00001	0.000288	0.000014		
LAM-MC-ICP-MS Mud Tank	2190	0.282523	0.000043	1.46718	0.00040	0.000105	0.000095	0.00455	0.00454

Table 2. Table 2 Hf Isotope Ratio Measurements, *TIMS results from Weidenbeck et al., [2]

Glitter Software Update

Dr. Norman Pearson

Macquarie University, Australia

Users of Glitter Software are advised to upgrade to the latest version of the software: Glitter 4.4.1. This latest release will read all data files produced from Agilent 4500 and 7500 instruments.

Availability:

Contact the Glitter Technical Support (technicalsupport@glittergemoc.com) to obtain Glitter 4.4.1.

This version release is available as a free upgrade but requires IDL 6.1 or higher. If you are running an older version of Glitter using IDL 5.X you will need to upgrade to IDL 6.X (for a nominal fee) from www.ittvis.com before installing the new version of Glitter.

New 68 Page Guide to Speciation Apps



This new handbook contains a collection of well referenced application briefs contributed by world wide authorities, covering a wide range of applications of hyphenated ICP-MS in elemental speciation.

Sections on the most common separation techniques are included, namely high-performance liquid chromatography (HPLC), which includes ion chromatography (IC), and gas chromatography (GC) as well as other separation techniques such as capillary electrophoresis (CE), field flow fractionation (FFF) and multi-MS techniques.

The Handbook is designed to show the breadth of work currently undertaken using hyphenated techniques involving ICP-MS – one of the fastest growing research and application areas in mass spectrometry.

More Information:

You can download a pdf of the Handbook of Hyphenated ICP-MS Applications, 5989-6160EN at: www.agilent.com/chem/icpms

Update on US EPA's Position on CRC-ICP-MS

Steve Wilbur Agilent Technologies, Inc., USA

Drinking water compliance monitoring using ICP-MS method 200.8.

The memo issued by the office of water (14 July, 2006) restates the position of the agency prohibiting the use of collision/reaction cell (CRC) technology to eliminate polyatomic interferences in the ICP-MS analysis of drinking water using Method 200.8 under the safe drinking water act. While the use of CRC equipped instruments is specifically permissible, they must be used with the cell turned off. The Office of Water in collaboration with the Office of Research and Development is currently completing a year long study with Agilent's support to evaluate the effectiveness of CRC ICP-MS for drinking water analysis.

Other (non-drinking water) compliance monitoring using CRC-ICP-MS.

The majority of non-drinking water compliance monitoring falls within two other EPA programs. The National Pollutant Discharge Elimination System (NPDES) and the Resource Conservation and Recovery Act (RCRA) regulate wastewater and solid waste handling in the US.

There is currently NO RESTRICTION on the use of CRC ICP-MS for compliance monitoring under either of these programs. In fact, in response to questions from Agilent on this issue, Shen-yi Yang – Inorganic Methods Program Manager at the EPA Office of Solid Waste in Washington DC, stated the following:

"...(while) reaction cell or collision cell technologies are not specifically outlined in the current version of Method 6020A, it is understood their application may have profound effects on eliminating interferences for trace level analyses, and this would be considered as an acceptable modification to Method 6020A as long as it can be demonstrated to be able to determine the analytes of concern in the matrix of concern at the level of concern to meet project-specific DQOs (data quality objectives)."

Her complete response can be read on the Agilent website: Go to www.agilent.com/chem/icpms Click the "ICP-MS Methods News" link under "Additional Information". John Bourbon, the Organic and Inorganic Chemistry Section Chief at Region 2 stated in an email response to Agilent on June 18, 2007 that:

"Region 2 considers the use of an ICP/MS instrument with collision/ reaction cell technology to be in compliance with USEPA Method 200.8 for the analysis of samples collected for NPDES compliance purposes, provided the requirements of USEPA Method 200.8, Section 9.2, Initial Demonstration of Performance, are satisfied. This assessment is consistent with the opinion of the USEPA Office of Water, Office of Science and Technology, Engineering and Analysis Division, responsible for the promulgation of approved analytical methods used for NPDES compliance monitoring."

His complete response can also be read on the Agilent website:

www.agilent.com/chem/icpms

Click the "ICP-MS Methods News" link under "Additional Information".

New ICP-MS ChemStation Rev - B.03.05

Tomo Yamada

Tokyo Analytical Division Agilent Technologies

New features of the latest revision of ICP-MS ChemStation - B.03.05 – include:

- Support for the 7500cx
- Helium mode semiquant factorsSupport for the High Matrix
- Introduction (HMI) kit • Control of the CETAC EXR-8
- Autosampler

Current rev.B.0x.0x users can upgrade to B.03.05 (for Windows XP only) free-of-charge from the Agilent ICP-MS website www.agilent.com/chem/icpms.

More information on upgrading your revision of ChemStation will be given in the next issue of the Agilent ICP-MS Journal.

21 CFR Part 11 Compliant Handling for ICP-MS ChemStation Data with Agilent's Enterprise Content Manager (ECM)

Katja Kornetzky

Solutions Manager Pharma QA/QC, Agilent Technologies, Waldbronn, Germany

Introduction

Data handling that is compliant with the FDA 21 CFR Part 11 guidelines is becoming more widespread. A major challenge for lab managers and IT professionals is to provide compliance solutions that do not impede productivity, but, in contrast, enable scientists and technicians to be even more efficient in their daily tasks.

Agilent has integrated the ICP-MS ChemStation (rev.B.03.06) with the OpenLAB Enterprise Content Manager (ECM) allowing Agilent 7500 users to store their ICP-MS ChemStation data in compliance with 21 CFR Part 11.

Operation of the ICP-MS ChemStation with ECM remains familiar, ensuring minimum disruption of current work practices.

What is OpenLAB ECM?

OpenLAB ECM is a web server based software platform that provides a secure, central repository for all kinds of data.

Rich content services allow users to capture, manage, collaborate, archive, and re-use all business critical information.

OpenLAB ECM: Not Just a Repository

The ICP-MS ChemStation, in combination with the User Access Control Pack and ECM Security, assures that data are automatically stored into ECM immediately after their creation. Once in ECM, the data are under audit trail and revision control.

• Users can access the Agilent OpenLAB ECM via a web client



Figure 1: Container-type storage of ICP-MS ChemStation data, including pdf-reports in ECM

and view data and pdf-reports from anywhere in the organization, see Figure 1.

- Access to the system is protected by a unique user name and password.
- Data can be searched and retrieved by using advanced search functions.
- Users can sign off on the data in the ECM web client using an ECM electronic signature.

ICP-MS ChemStation/ECM Integration Ensures:

• Data security by:

- Mandatory logon with a legitimate Windows user-ID/password combination, both for ICP-MS ChemStation and OpenLAB ECM
- Configurable password policies such as renewal frequency or minimum length
- Configurable account lock-out policy

• Data integrity by:

- Automatic data storage and versioning
- Full version control for ALL relevant data including original result reports in pdf format
- Integrated archival and longterm storage in a content

Electronic Signature							
- User information							
Ilsemame: supervisor	Sign						
	Connel						
Password:	Lancer						
Domain name: BUILT-IN							
Signed-at location: Waldbronn							
Reason for signing							
Default reason: I am approving this document.							
C User specified:							

management system

- Data traceability by:
- Automatic user-independent audit trail
- Electronic signatures, see Figure 2.

Data Sharing Within Your Lab or Across the Enterprise

Agilent OpenLAB ECM is available in a workgroup version for up to 5 users in a single laboratory or as a full-fledged enterprise system for an unlimited number of users.

Equipment Validation Services for 7500

Of course compliance is more than data management and Agilent has a full suite of products that combine to make a full solution. New Equipment Qualification Installation Qualification/ IQ) and Operational Qualification/ Performance Verification (OQ/PV) are available not only for the Agilent 7500 ICP-MS System but also the ICP-MS ChemStation, and ICP-MS ChemStation with ECM.

More Information

Agilent Application Note: OpenLAB ECM - Workgroup Through Enterprise, Pub Number 5989-6978EN.

> Agilent Technical Note: Agilent ICP-MS ChemStation – Complying with 21 CFR Part 11, Pub Number 5989-4850EN.

Figure 2: Traceability: eSignatures can be applied to ICP-MS ChemStation data in OpenLAB ECM

Anniversary Celebrations: 20 Years in ICP-MS

Tomo Yamada Tokyo Analytical Division Agilent Technologies



The forerunner of the 7500 Series was introduced in Japan in 1987 by Yokogawa (original ICP-MS division of Agilent Technologies). To celebrate this landmark, Agilent's Tokyo Analytical Division (TAD) hosted a 20th Anniversary Seminar attended by more than 120 customers and invited guests.

Special thanks go to speakers Prof. Haraguchi (Nagoya Univ.), Prof. Furuta (Chuo Univ.), Dr. Nonose (AIST) and presenters from Kanto Chemicals and EMATEC Kansai.

Olympic Gold for 2000th 7500 Series ICP-MS

Tomo Yamada

Tokyo Analytical Division Agilent Technologies

An Official Supporter and the exclusive metals supplier of every gold, silver and bronze medal awarded at the Vancouver 2010 Olympic and Paralympic Winter Games - Teck Cominco Metals Ltd - has taken delivery of the 2000th Agilent 7500 Series ICP-MS to ship from Agilent's Tokyo factory. Teck Cominco is a diversified mining and metals company, headquartered in Vancouver, Canada. and is known as a world leader in the production of zinc and metallurgical coal and a significant producer of copper, gold and specialty metals. www.teckcominco.com

This information is subject to change without notice

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New Agilent ICP-MS Users

A very warm welcome to all companies and institutions that have recently added an Agilent ICP-MS to their analytical facilities. Remember to join the Agilent web-based ICP-MS User Forum - the place where you can exchange information relating to your new ICP-MS. You will also find a host of resources in the Forum designed to help you get the most from your 7500.

To access the Forum, you will simply need to log-in to the Agilent web site, or register if you haven't already, and enter your instrument's serial number on your first visit only. Look for the link to the ICP-MS User Forum from: www.agilent.com/chem/icpms

4500 Series EOS Oct 31, 2007

A reminder that Agilent will continue to fully support all 4500 Series instruments until their End of Support (EOS) date of October 31, 2007.

Contact your Agilent support sales representative for more information.

Trade Shows and Conferences

Agilent ICP-MS Workshop, Massy, France. October 16, 2007 BCEIA Beijing, PRC October 18 - 21, 2007 Agilent ICP-MS User Meeting, Jyvaskyla, Finland. October 22-23, 2007 Anacon-Analytica, Hyderabad, India October 31 - November 2, 2007 International Symposium on Metallomics (ISM), Nagoya, Japan, November 28 - December 1, 2007 Agilent ICP MS Workshop, St. Potersburg, Puscia, November 12, 14, 2007

Agilent ICP-MS Workshop, St Petersburg, Russia. November 13-14, 2007 **2008 Winter Conference on Plasma Spectrochemistry** January 7-12, 2008, Temecula, CA, USA

Agilent ICP-MS Users Meeting at 2008 WPC

Location: The Pechanga Resort & Casino, 45000 Pechanga Parkway, Temecula, USA

All Agilent ICP-MS users attending the 2008 Winter Plasma Conference are invited to join Agilent's ICP-MS User Meeting on **Tuesday January 9th, 2008, from 7:00pm** - **9:00pm** in the Conference hotel.

This annual event offers the opportunity to get the latest ICP-MS information and network with fellow users. It's a great opportunity to field some questions, and get some answers from Agilent's team of ICP-MS experts.

Register on line to secure a place: www.agilent.com/chem/icpmsuser

Agilent ICP-MS Publications

To view and download these latest publications, go to www.agilent.com/chem/icpms and look under "Library Information"

- Application: Handbook of Hyphenated ICP-MS Applications, 5989-6160EN
- Application note: Determination of Organic and Inorganic Selenium Species Using HPLC-ICP-MS, 5989-7073EN
- Application note: Measurement of Trace Elements in Malt Spirit Beverages (Whisky) by 7500cx ICP-MS, 5989-7214EN

Front page photo:

Agilent's Katsuo Mizobuchi, Senior ICP-MS Applications Engineer and specialist in semiconductor applications, based in Tokyo Japan.

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