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Direct Analysis of Undiluted Soil Digests by HMI-ICP-MS

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Introduction

Agilent has developed the High Matrix Introduction (HMI) accessory for ICP-MS as an alternative to conventional dilution. The HMI modifies the sample introduction system of the Agilent 7500 Octopole Reaction System (ORS) ICP-MS, making it possible to directly measure sample solutions with total dissolved solids (TDS) exceeding 1%. In collaboration with the Eurofins Analytico laboratory in the Netherlands, a 7500cx/HMI was used to directly measure high TDS soil extracts in compliance with Dutch regulatory guidelines pertaining to contaminated soils (AS3000).

Direct Analysis of Aqua Regia Digests of Soils Using HMI-ICP-MS

Due to the high TDS levels and high acid concentrations of digested soils, dilution is necessary prior to analysis by ICP-MS. Analytico currently uses the Agilent Integrated Sample Introduction System (ISIS) to perform online dilutions to meet these requirements. However, the limitations of conventional dilutions prompted Analytico to evaluate the potential of HMI as a faster, simpler, less expensive replacement for conventional autodilution.

Instrumentation

An Agilent 7500cx ICP-MS was equipped with the HMI. A Burgener MiraMist nebulizer was used. The plasma was optimized in ultra robust mode with maximum aerosol dilution. All analytes except selenium were acquired in helium collision mode, thus eliminating the need for no-gas mode and the associated time required for mode switching. Selenium was acquired in hydrogen reaction mode due to the low detection limits required.

Analyte - isotope	ORS mode	MDL (1) (mg/kg)	MDL (2) (mg/kg)	Dutch Required MDL (mg/kg)
Be 9	He	0.042	0.046	0.1
V 51	He	0.255	0.481	1
Cr 52	He	2.300	4.517	15
Co 59	He	0.147	0.348	1
Ni 60	He	0.770	0.922	3
Cu 63	He	0.502	1.303	5
Zn 66	He	1.704	3.104	17
As 75	He	0.549	1.079	4
Se 78	H2	0.832	2.041	10
Se 78	He	1.064	1.991	10
Mo 95	He	0.195	0.413	1.5
Ag 107	He	0.278	0.701	1
Cd 114	He	0.058	0.066	0.17
Sn 118	He	0.589	1.353	6
Sb 121	He	0.333	0.401	1
Te 125	He	1.217	2.112	10
Ba 135	He	3.041	6.227	15
Hg 201	He	0.014	0.025	0.05
TI 203	He	0.285	0.546	3
Pb 208	He	1.197	2.844	13

Table 1. Calculated Method Detection Limits (mg/kg) According to the Requirements of Dutch Regulation AS3000

 $MDL(1) = 3\sigma$ of 10 replicates taken on the same day

 $MDL(2) = 3\sigma$ of 10 replicates taken on different days – information only

Sample Preparation

Sample Analysis

Actual soil samples received from Analytico's customers were prepared by adding 1 gram of soil to 8 mL of aqua regia prior to microwave digestion. Digested samples were then diluted to 50 mL final volume in ultra pure (18.2 M Ω) water. The final acid concentration is 4% HNO₃ and 12% HCl.

Calculation of Method Detection Limits (MDLs)

MDLs were calculated as 3-sigma of 10 replicates of a low-level (between one to three times the required MDL) spiked sand sample measured consecutively and also measured on 10 different days over a 30-day period (Table 1). For all regulated elements, the calculated MDLs exceeded the Dutch regulatory requirements by nearly an order of magnitude or better. So even with the HMI operating at maximum effective dilution, the system has ample sensitivity for the application. In order to test the long-term robustness of the HMI-equipped system, a 23-hour sequence consisting of an initial calibration and 235 soil samples was analyzed. Absolute drift was measured by monitoring the recovery of the five internal standards (6Li, Ge, Rh, In, Ir) in both gas modes (He and H_2) over the course of the sequence. Normalized recoveries (relative to the method blank) are shown in Figure 1. Overall downward drift over 23 hours was approximately 20%, which is easily corrected by internal standards and not sufficient to have a detrimental effect on method accuracy or sensitivity. By comparison, a similar system without HMI, running these samples directly would suffer severe loss of sensitivity (> 80%) before the sequence was completed.

AS3000 requires continuing calibration verification (CCV) samples (1 mg/kg As, 50 mg/kg Hg, remaining elements 2 mg/kg) to be analyzed



Figure 1. Normalized internal standard recovery in digested soil samples for the duration of the 23-hour sequence. Numbers next to each ISTD in the caption denote the ORS gas mode (#2 = H2, #3 =He).



Figure 2. Continuing calibration check (CCV) recoveries (n-21) for all analyte elements except Hg and As, over the 235-sample sequence of soils and sludges. Method required control limits (90 to 110%) are shown in red. Numbers after element names indicate ORS mode, #2 – H2 mode, #3 – He mode.

after each 12 soil samples. Results are shown in Figure 2. Acceptable recoveries must be within \pm 10%. Ten percent control limits are shown in red, indicating that all CCV recoveries were well within the prescribed limits for the entire sequence. This excellent calibration stability not only ensures the most accurate sample results, but also eliminates time wasted in unnecessary recalibrations should a CCV fail during the sequence.

Productivity

The operation of the HMI accessory does not adversely affect productivity in any way, since the HMI conditions are constant throughout operation and do not require any additional execution or stabilization time. Therefore, a given method run with HMI would take the same amount of time per sample as one run without HMI. HMI is also fully compatible with Agilent's time saving pre-emptive and intelligent rinse functions, which minimize time wasted during both sample uptake and rinseout. Furthermore, since HMI permits the direct analysis of undiluted samples of many types, the extra time associated with either manual or autodilution is saved, which considerably shortens the total time (prep plus analysis) required per sample. The improved stability as a result of HMI use can also minimize the need for recalibrations and sample reruns, further reducing the average run-to-run time. In this work, the average run time for a sample in a 23-hour, 235-sample sequence of undiluted soil digestates was 5.9 minutes, including acquisition in both H_2 and He modes.

Conclusions

Based on Analytico's evaluation, when compared with conventional autodilution for high TDS, Agilent's HMI interface provided a number of significant advantages:

• Speed – HMI does not require liquid dilution of sample and stabilization of diluted sample. It also permits the use of Agilent's pre-emptive rinse function, which allows rinsing of the sample tubing to begin before acquisition has finished.

- Low maintenance no tubing to replace and no moving parts to maintain.
- Simple no critical timing issues or plumbing common to continuous flow autodilutors.
- Flexibility since hardware changes or reconfigurations are not required after installation of HMI, the system can be switched between conventional mode and HMI mode on the fly.

These advantages have allowed Analytico to use a single 7500cx ICP-MS fitted with HMI to replace several instruments required for the analysis of these sample types, including conventional ICP-MS, ICP-OES, and a dedicated mercury analyzer.

Further Information

Agilent Application Note: Direct Analysis of Undiluted Soil Digests Using the Agilent High Matrix Introduction Accessory with the 7500cx ICP-MS, 5989-7929EN

Improved MDLs in High Matrix Samples using HMI-ICP-MS

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Objective

TestAmerica Savannah's goal for the High Matrix Introduction (HMI) sampling accessory with the 7500ce ICP-MS is to be able to analyze high matrix environmental samples especially soils and Toxicity Characteristic Leaching Procedure (TCLP) extracts without the complications of a dilution step while maintaining Method Detection Limit/Reporting Limit (MDL/RL) at current levels.

The Company

TestAmerica Savannah was founded in 1975 and employs over 100 environmental professionals and support personnel in a state-of-the-art 55,000 square foot facility. The company has built up a reputation nationwide for consistent quality performance at competitive prices by specializing in custom project management services.

Analytical Challenges

Traditionally we dilute all of the digested samples we introduce to the ICP-MS by a factor of 1/5 for liquids and 1/10 for soils to reduce the matrix effects contributed by both the acids used in the preparation and the samples themselves. There are several disadvantages associated with this practice:

- Possibility of introducing contamination from the pipette tips or the diluent
- Chance of "human" error of simply preparing an inaccurate dilution (rare)
- Sample prep time and cost of reagents

HMI Methodology

With the HMI unit installed on the 7500ce (upgraded from a 7500c) we are now able to introduce samples of any matrix to the ICP-MS directly, without a dilution step, and thus eliminate the disadvantages associated with conventional dilution.

In contrast to conventional dilution, the HMI uses "aerosol" dilution to introduce a less concentrated sample aerosol into the plasma of the ICP-MS thereby reducing sample load and minimizing matrix effects and signal suppression.

High Acid Matrices

A significant hurdle to running undiluted environmental samples is related to the final acid concentration specified in USEPA method 3050B for soils and wastes using the "hot-plate" procedure, which includes 10% HCl. Method 3010A for liquids specifies a final concentration of 5% HCl. Prior to using the HMI, matrix matching during the dilution step was used to normalize the acid concentration in standards and both types of samples. With HMI, we opted for a standard matrix of 5% HNO₃ and 5% HCl for standards and used the online internal standard channel to dilute 1:1. which reduced the HCl concentration to 5% in the soil samples. This was done in order to compensate for the higher than normal efficiency of the PFA-100 nebulizer that we prefer to use. The HMI was set-up for typical "ultrarobust" analysis and maximum aerosol dilution. Table 1 shows comparison MDL data (mg/kg) for soils with and without the HMI. With the HMI, the digestates were analyzed directly (undiluted); without the HMI, the digestates were diluted 1/10. The results highlight an improvement in MDLs for the majority of elements when the HMI is used.

Reproducible Results

To test the precision and accuracy of the HMI results, samples were analyzed with associated Quality Control (QC) samples according to methods 6020A and 200.8. A serial dilution and post-digestion spike were analyzed to test the effectiveness of the HMI at reducing matrix effects (Table 2). Laboratory Control Spikes and Spike Duplicates (LCS/LCSD) Matrix Spike and Spike and Duplicates (MS/MSD) were also analyzed to assure accuracy in the procedure and the results. All elements were within specified control limits.

	нмі	w/o HMI MDL (1/10 dil.)	
Analyte / mass	MDL		
	mg/kg	mg/kg	
AI / 27	4.1	5.8	
Sb / 121	0.026	0.031	
As / 75	0.03	0.15	
Ba / 137	0.053	0.17	
Be / 9	0.021	0.018	
B/ 11	0.33	1.3	
Cd / 111	0.029	0.031	
Ca / 40	5.7	8.4	
Cr / 52	0.031	0.13	
Co / 59	0.0064	0.0094	
Cu / 63	0.078	0.14	
Fe / 56	1.7	4.6	
Pb / 208	0.055	0.03	
Mg / 24	1.3	1.9	
Mn / 55	0.037	0.11	
Mo / 95	0.036	0.058	
Ni / 60	0.024	0.036	
K/ 39	3.8	6.5	
Se / 78	0.13	0.044	
Ag / 107	0.005	0.0071	
Na / 23	1.8	15	
Sr / 88	0.051	0.066	
TI / 205	0.026	0.015	
Sn / 118	0.105	0.24	
Ti / 47	0.17	0.12	
V / 51	0.041	0.096	
Zn / 66	0.84	0.64	

Table 1. Comparison of MDL for a suite of elements using HMI and normal dilution.

Conclusions

The addition of the High Matrix Introduction System to the Agilent 7500ce ICP-MS has significantly improved the productivity while reducing the cost associated with analyzing both soils and waters according to EPA methodologies. Performance including compliance with all QA/QC requirements is equal to or better than the standard (no HMI) system and detection limits are slightly improved.

Analyte	Sample	Serial Dil.		Post-Spike	
	ug/L @ instrument		RPD (%)	(ug/L @ instrument)	Post-Spike rec. (%)
Aluminium / 27	124300	26750	7.3	123000	NC
Antimony / 121	2.377	0.8908	NC	22.59	101
Arsenic / 75	39.26	9.552	19.5	59.66	102
Barium / 137	1153	231.4	0.3	1180	NC
Beryllium / 9	6.402	1.315	2.7	26.31	100
Cadmium / 111	2.073	0.4975	18.2	21.07	95
Calcium / 40	30320	6477	6.6	31990	84
Chromium / 52	182.7	38.5	5.2	202	97
Cobalt / 59	81.52	17.43	6.7	101.3	99
Copper / 63	105.3	22.08	4.7	125.5	101
Iron / 56	162800	35320	8.1	163000	10
Lead / 208	76.63	15.6	1.8	96.46	99
Magnesium / 24	28860	6308	8.9	30360	75
Manganese / 55	8311	1818	9.0	8563	NC
Molybdenum / 95	5.722	1.107	3.3	26.79	105
Nickel / 60	156.6	34.62	10.0	177.2	103
Potassium / 39	10780	2302	6.6	12910	107
Selenium / 78	1.081	0.1919	11.9	18.38	86
Silver / 107	0.3305	0.06239	5.8	20.62	101
Sodium / 23	3716	797.2	7.0	5543	91
Strontium / 88	233.8	53.62	13.7	268.8	NC
Thallium / 205	1.707	0.2635	NC	5.631	98
Tin / 118	21.55	4.123	4.4	43.79	111
Titanium / 47	5848	1232	5.2	5924	NC
Vanadium / 51	405	86.42	6.5	426.1	106
Zinc / 66	331.1	86.18	26.2	353.8	114

Table 2. Serial dilution (1/5) precision shown as Relative Percent Difference (RPD) and post digestion spike recoveries. Spike recoveries are not calculated (NC) when the spike amount is less than 25% of the original sample concentration.

Australia's Eighth Agilent ICP-MS User Meeting

Agilent's commitment to the power of user meetings was discussed in issue 29 of the ICP-MS Journal in November 2006. The highly successful event "down under" is an excellent example of this philosophy.

The Eighth Annual Australian Agilent ICP-MS User Meeting was held on 22^{nd} and 23^{rd} April 2008 in Perth, Western Australia. The meeting was hosted by Prof John Watling and his group in University of Western Australia. Fred Fryer, the senior ICP-MS applications specialist in South East Asia, was an excellent Master of Ceremonies throughout the proceedings. Most of the talks were contributed by users and covered

topics as diverse as forensic studies, "imaging" the distribution of metals in rat brain, improving sample throughput in a busy clinical lab and fundamental laser ablation investigations. There was ample time for our customers to provide frank feedback in the many Q&A sessions over the two days as well as at the very enjoyable dinner that was held in "Little Creatures" a restaurant/ microbrewery in historic Fremantle.

The group of 24 users and 4 Agilent staff included veterans as well as some new faces, and the meeting was, as usual, a useful forum for swapping ideas and operating tips and tricks. We owe a special thanks to John Watling (UWA), David Bishop (UTS), Alison Beavis (UTS), Vesna Dolic (Glass Expansion), Cameron Scudding (TSW Analytical), Matthew Baxter (Sullivan & Nicolaides), Greg O'Neill (South Australia Water), Leonid Danyushevski (University of Tasmania), Tamara Pilgrim (TSW Analytical) and Mike Whitby for their excellent contributions. Feedback on the user meeting was universally positive and all are looking forward to the 2009 event which will be held in Tasmania next March.



Analysis of Trace Contaminants in High-Purity Metals by HMI-ICP-MS

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Introduction

High-purity metals analysis is challenging due to the very low detection limits required in the presence of a very high matrix concentration. Additionally, the use of aggressive acid mixes can be damaging to the ICP-MS interface. In this work, high-purity metal certified reference materials (CRMs) JSAC 01211 aluminum and NIST 3982 copper were analyzed using an Agilent 7500cx fitted with the HMI accessory. Samples were prepared by gently dissolving 1 gram of sample into 100 mL of 20% aquaregia or 10% nitric acid and analyzed directly. Results for CRM JSS003-53 iron are included in ref [1].

Note that even when analyzing 20% aquaregia, nickel sampling and skimmer cones were used.

Recoveries of the certified values are shown in Tables 1 and 2. Long-term stability was tested by repeatedly (n = 150) analyzing a 1% Cu solution

Element (m/z)	Cell Gas	Certified value	Result	
		mg/kg	mg/kg	Recovery (%)
Mg (24)	He	2.82 ± 0.13	2.841	101
Ti (47)	He	1.96 ± 0.07	2.073	106
Cr (52)	He	1.13 ± 0.06	1.098	97
Mn (55)	He	1.73 ± 0.06	1.730	100
Fe (56)	He	9.4 ± 0.3	10.016	107
Cu (63)	He	3.48 ± 0.11	3.738	107
Zn (68)	He	2.03 ± 0.13	2.117	104
Zr (90)	He	2.02 ± 0.13	2.000	99

Table 1. Recovery of Certified Values for JSAC 0121 (High-Purity Aluminum)

Element (m/z)	Cell Gas	Certified value	Result	
		mg/kg	mg/kg	Recovery (%)
Fe (56)	He	11.4 ± 0.5	11.52	101
Ni (60)	He	7.0 ± 0.1	7.04	101
Zn (68)	He	24 ± 1	24.18	101
As (75)	He	25.0 ± 3	27.62	110
Se (78)	He	17.5 ± 0.8	17.33	99
Ag (107)	He	20.1 ± 0.2	19.75	98
Sn (118)	He	4.8 ± 0.6	5.46	114
Sb (121)	He	7.5 ± 0.1	7.25	97
Te (125)	He	10.1 ± 0.2	10.12	100
Pb (208)	He	9.9 ± 0.6	10.15	103
Bi (209)	He	2.0 ± 0.3	2.09	105

Table 2. Recovery of Certified Values for NIST 398 (Unalloyed Copper)

in 10% nitric acid and monitoring internal standard recovery (see Figure 1).

HMI Advantage

Using the HMI accessory with the Agilent 7500cx permits simple, direct analysis of high TDS, high acid metal digests, without prior dilution. Results for the standard reference high-purity metals showed excellent agreement with certified values.

Long-term stability was excellent as evidenced by stable internal standard recoveries over a 150-sample sequence of 1% Cu solutions.

More Information

1. Agilent Application Note: Using the Agilent 7500cx ICP-MS with High Matrix Introduction Accessory for the Analysis of Metals in High Tech Manufacturing, 5989-8095EN



Figure 1. Internal standard signal stability over 150-sample sequence - 1% copper solution in 10% nitric acid.

New Revision (B.03.07) of ICP-MS ChemStation

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The latest revision of the ICP-MS ChemStation software (B.03.07) improves the integration of add-on hardware and software products, such as the High Matrix Introduction (HMI) accessory, the OpenLAB Enterprise Content Manager (ECM) and the GC/LC ChemStation.

Improved HMI User Interface and Functionality

The High Matrix Introduction (HMI) accessory for the Agilent 7500cx enables analysts to directly measure samples with total dissolved solids (TDS) content of 1% or higher (no other ICP-MS can do this!). The HMI software has now been improved for easier set-up and use. The new user interface enables users to select a preset condition from a single screen. Furthermore, this new revision provides the flexibility to switch tuning files even after a preset condition is loaded.

Improved Security with ECM Integration

For users of the ICP-MS ChemStation ECM Integration in combination with the ICP-MS User Access Control Pack for 21 CFR Part 11 compliance, access security capability has been enhanced. The "Lock/ Unlock ChemStation" function is available when using with the User Access Control Pack (G3263A). In addition, operator change over is easy – protecting ICP-MS ChemStation from unauthorized operation.

Better Hyphenated ICP-MS Applications with GC and LC

The Agilent GC/LC ChemStation rev.B.03.02 can now be installed together with the ICP-MS ChemStation on the same PC. (Note: English versions only). A method from the LC/GC ChemStation can be run as part of an ICP-MS ChemStation sequence. Also, B.03.07 supports the GC injector in the back position as well as the front position (the previous revision only supported the front injector). Note: Dual injection is not supported.

Upgrading Your ICP-MS ChemStation

For current rev.B.0x.0x users, a free upgrade to B.03.07 (for Windows XP users) can be downloaded from the Agilent ICP-MS website www.agilent.com/chem/icpms. ("ICP-MS ChemStation Updates & Downloads" in the "ICP-MS Software" section).

More Information

- High Matrix Introduction Accessory: www.agilent.com/chem/hmi
- Application Note: Agilent ICP-MS ChemStation – Complying with 21 CFR Part 11, 5989-4850EN

Reference Materials Web Resource

Any users looking for information on inorganic reference materials (RM) may find "The Database of Natural Matrix Reference Materials" Compiled of use. by the International Atomic Energy Agency (IAEA), the database contains information on over 1751 RM relevant to trace element content (plus other constituents) of biological, food and environmental materials of natural origin.

"The Database of Natural Matrix Reference Materials" is freely accessible from: www-naweb.iaea.org/nahu/

nmrm/nmrm2003/index.htm

ICP-MS Supplies Quick-Reference List

Rich Quashne

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www.agilent.com/chem/quick-icp-ms

Order directly from Agilent online, from your local Agilent representative, or through an authorized Agilent distributor.

Free ICP-MS e-Seminars

Agilent has organized three e-seminars designed to keep you up-to-date with the latest technological and application developments in ICP-MS.

In the first e-Seminar you can learn how Agilent's new High Matrix Introduction (HMI) accessory can permit the direct analysis of high total dissolved solids ICP-OES samples directly by ICP-MS. The second presentation discusses regulatory pertaining requirements to environmental analysis. The final seminar explains how life science researchers use ICP MS to locate and quantify elementally labeled biomolecules or measure heteroatoms like sulfur and phosphorus in proteins and peptides.

e-Seminar: Extending ICP-MS into the Realm of ICP-OES – The Agilent High Matrix Introduction Accessory Presenter: Steve Wilbur, Senior Applications Scientist, Agilent Technologies, Inc. Available as a recorded event

e-Seminar: Environmental ICP-MS -EPA Methods Update Presenter: Steve Wilbur, Senior Applications Scientist, Agilent Technologies, Inc. Available as a recorded event

e-Seminar: ICP-MS – The Role of Elemental Mass Spectrometry in Life Sciences Research Presenter: Dr. Joseph Caruso, University of Cincinnati Date: May 8, 2008, 1:00 pm - 2:00 pm US Eastern Std Time

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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 done so previously, 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 Reaches EOS

The Agilent 4500 Series ICP-MS has reached its End of Support (EOS). For more information on transitioning to the 7500 Series, contact your Agilent support sales representative or visit: **www.agilent.com/chem/icpms**

Agilent Food Safety and Environmental Testing Seminar Series Dates: April 15-May 22, 2008

Location: Various US cities

The Inorganic session of this two stream event will discuss the New Heonly 7500cx ICP-MS and HMI accessory as well as numerous hardware and software improvements on the best selling Agilent 7500 Series. We will also provide an update on all current EPA methods for ICP-MS.

www.agilent.com/chem/quantifytour

Trade Shows and Conferences

4th International Conference on Trace Element Speciation in Biomedical, Nutritional and Environmental Sciences May 25-29, 2008, Munich, Germany www.gsf.de/spec

ASMS June 1-5, 2008, Colorado Convention Center, Denver, Colorado, USA www.asms.org

Nordic Conference on Plasma Spectrochemistry June 15-18, 2008, Loen, Norway www.nordicplasma.com

Agilent ICP-MS Publications

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

- Performance Characteristics of the Agilent High Matrix Sample Introduction (HMI) System for the 7500 Series ICP-MS, 5989-7737EN
- Application: Direct Analysis of Undiluted Soil Digests Using the Agilent High Matrix Introduction Accessory with the 7500cx ICP-MS, 5989-7929EN
- Application: Using the Agilent 7500cx ICP-MS with High Matrix Introduction Accessory for the Analysis of Metals in High Tech Manufacturing, 5989-8095EN
- Application: Multi-Element Determination of Heavy Metals in Dietary Supplements Using Collision/Reaction Cell ICP-MS, 5989-7959EN

Front page photo:

Agilent China's ICP-MS team members (L to R): Product Specialists Wang Wenqi & Song JuanE, and Application Engineer Jing Miao. Separate photo: Chen Cheng-Xiang, Product Specialist

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