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Agilent Technologies

New Japanese Regulations/ Analytical Method for Drinking Water

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More than 200 Agilent ICP-MS systems have already been installed in Japan for various environmental analyses, and a substantial number of these systems are used for drinking water or related sample analysis. ICP-MS is contributing to the efficient analysis of trace metals in drinking water.

Brief History and Current Status of Japanese Drinking Water Supply

The Japan Water Supply Act was first promulgated in 1957 and the Quality Standard for Drinking Water was set in the following year. Since then, drinking water has been supplied safely by measuring water quality throughout the nation. Currently, more than 96% of the total population is utilizing public drinking water. Recently, even greater attention has been given to the quality of the drinking water due to the

occurrence of pathogenic microorganisms and contamination by chemicals in the water supply.

The Quality Standard for Drinking Water in Japan

With respect to regulatory requirements, the Quality Standard is classified into the following three categories:

1. Items related to the minimum requirements needed to maintain "basic" quality
2. Items related to "comfortable" quality as a goal of higher-quality drinking water
3. Items related to "monitoring" quality for suspicious elements related to possible risk.

The Quality Standard currently includes 17 metals (see Table 1). Since many of these metals are toxic (and some are potentially carcinogenic), the maximum allowable concentrations (MACs) are set particularly low. It must be noted that the quantification limits of the techniques used to measure these elements must be below 1/10 of the MACs to ensure complete confidence in the result.

In 2001, the "drinking water test method" was revised and expanded the use of ICP-MS to 14 of the regulated elements. Measurements using ICP-OES instruments had been previously approved, however

this required sample pre-concentration or other sample preparation techniques for many of the elements to ensure compliance with the LOD requirements. The reduction of the MAC for many of the more toxic elements has placed more pressure on the older ICP-OES method. Undertaking measurements using ICP-MS systems eliminates the need for the complicated and time-consuming sample preparation and the ICP-MS method is expected to rapidly replace the older one based on ICP-OES. Moreover, ICP-MS is the only commonly used technique that can easily measure uranium directly at 0.2ppb - 1/10 of the MAC of uranium, which is currently set at 2 ppb.

Due to government deregulation in 2001, it has now become possible for third parties to perform drinking water analysis. Therefore, most small- or medium-scale water supply facilities are now able to outsource analyses to analytical service labs. At the same time, such analytical labs must now assume responsibility for their quality control on measurements in order to ensure reliable data. The 2001 revision to the Drinking Water Test Method requires the establishment of a Standard Operating Procedure (SOP) for an analytical method and performance of the analysis according to the SOP.

(continued on page 3)

Control Elements	Quality Standard (ppb)	ICP-OES	ICP-MS	Note
B	200	See note	Yes	Detection limit of ICP-OES is more than 60 ppb*
Na	200000	Yes	-	Can be done using Agilent 7500 ICP-MS**
Al	200	See note	Yes	Ultrasonic Nebulizer required for ICP-OES
Cr	50	See note	Yes	Ultrasonic Nebulizer required for ICP-OES
Mn	50	Yes	Yes	
Fe	300	Yes	-	Agilent 7500c ICP-MS easily achieves 1/100**
Ni	10	See note	Yes	Ultrasonic Nebulizer required for ICP-OES
Cu	1000	Yes	Yes	
Zn	1000	Yes	Yes	
As	10	See note	Yes	Hydride Generator required for ICP-OES
Se	10	See note	Yes	Hydride Generator required for ICP-OES
Mo	70	See note	Yes	Ultrasonic Nebulizer required for ICP-OES
Cd	10	See note	Yes	Ultrasonic Nebulizer required for ICP-OES
Sb	2	See note	Yes	Hydride Generator required for ICP-OES
Hg	0.5	-	-	Can be done using Agilent 7500 ICP-MS**
Pb	50	See note	Yes	Ultrasonic Nebulizer required for ICP-OES
U	2	See note	Yes	Column Conc + Ultrasonic Neb* required for ICP-OES

Table 1 Regulated Metals in Drinking Water

*Described in the 2001 revision of Drinking Water Test Method for the Determination of 1/10 of maximum allowable concentration

** Observation using Agilent 7500 Series ICP-MS

- No description in the 2001 revision of the Drinking Water Test Method

New Japanese Regulations/Analytical Method for Drinking Water

(continued from page 2)

Contribution of Agilent 7500 Series ICP-MS

The newly introduced ICP-MS system, the Agilent 7500c, has a reaction cell and a wide dynamic range. Combined, these features provide a perfect solution for the analysis of all the metals required under the 2001 revision of the Drinking Water Test Method, including the analysis of Fe and Na, as well as other trace metals such as As and Se that were previously considered difficult to measure using conventional ICP-MS.

Agilent Technologies recently introduced the enhanced Quality Control (QC) template for its Intelligent Sequence QA/QC (Quality Assurance/Quality Control) software in compliance with the Japanese Drinking Water Test Method and associated SOP, see Figure 1. The template and SOP provide easy set-up for Quality Control of drinking water analysis.

The calibration standards, quality control solutions and blanks are placed in pre-defined positions of the autosampler and the entire analysis sequence is performed automatically according to the QC template. This automated method includes: checking instrument tuning, creation of calibration curves, verification of calibration curves, measuring samples, checking instrument working condition, and monitoring of measurement errors.

If an error is detected, a flag will be reported and the measurement can be repeated depending on the pre-configured setup in the Intelligent Sequencing QA/QC software. Various real-time reports can be generated including those associated with QC, calibration curves and results. It is easy for users to modify the QC template in order to meet the wide range of user-specific requirements worldwide, not limited simply to testing of Japanese drinking water.

It is extremely important that the quality of drinking water be carefully and thoroughly monitored and tested to ensure that it meets the highest quality standard. The Agilent 7500 Series ICP-MS with its Intelligent Sequence QA/QC software is an efficient analytical tool that allows for easy and high quality analysis of trace metals in drinking water, enhancing the productivity of routine analytical work.

Update on US EPA Arsenic Rule for Drinking Water

After considerable controversy, the US EPA has finally agreed to implement a 10 ppb Standard for Arsenic in Drinking Water (EPA 815-F-01-010, October 2001). This standard will set the maximum contaminant level (MCL) for arsenic in drinking water at 10 ppb. All US drinking water systems regulated by this MCL will need to comply

by Jan 2006. Of the 74,000 systems currently regulated by the MCL, approximately 4000 will need to install additional treatment or other measures to comply.

It is estimated that reducing the MCL from 50 ppb (established by EPA in 1975), to 10 ppb will prevent approximately 40-60 cases of bladder and lung cancer per year and reduce other diseases such as diabetes and heart disease.

A previous proposal, June 2000, recommended lowering the arsenic MCL to 5 ppb. This was to be finalized by June 2001 but was replaced by the current ruling.

Significantly, the new ruling will also withdraw the two ICP-AES methods (EPA 200.7 and SM 3120B) which are allowed for measurement at the old 50 ppb limit, citing insufficient sensitivity. Approved methods will include two graphite furnace and one hydride AA method and ICP-MS (EPA 200.8).

Details are available at: <http://www.epa.gov/OGWDW/new.html>

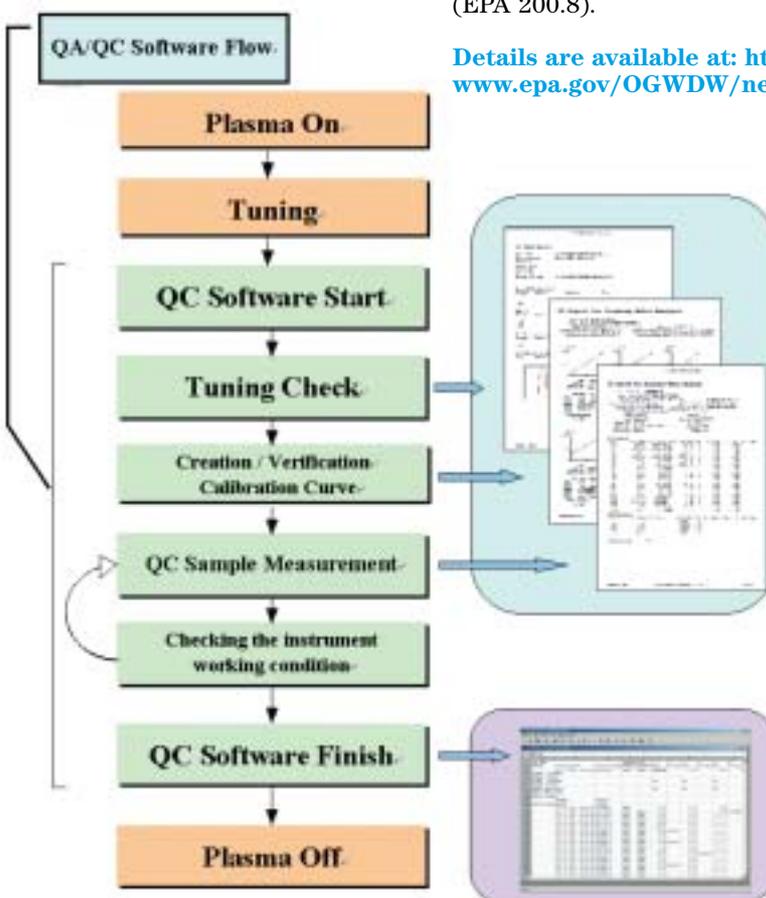


Figure 1. QA/QC Software flow and outputs. Various real-time reports can be generated including those associated with QC, calibration curves and results. Finally a list of analysis results is available.

Customer Profile - Severn Trent Laboratories

Quoted as the UK's leading environmental testing laboratory and the largest environmental laboratory company in the USA, Severn Trent Laboratories (STL) UK, took delivery of their second 7500i ICP-MS in February 2002. Based in Bridgend, South Wales, UK, the lab analyses over 1500 drinking water samples every week.

There has been a long tradition of drinking water analysis at the Bridgend lab, which was initially set up as one of the Welsh Water group of companies. During this period, soils and effluents as well as drinking water samples were analysed at Bridgend. In April 2000, the lab was acquired by Severn Trent Laboratories and over an integration period of 18 months it became the centre of excellence for drinking water analysis. With samples from all over the UK being directed to South Wales for analysis, the lab is currently the largest facility in the world for chemical analysis.

STL supply their clients with clean sample containers and clear sampling guidelines. Once at STL, samples are registered and managed through a sophisticated central computer system. Drinking water samples requiring analysis by ICP-MS are then loaded onto a roller-style conveyor belt, which carries the samples to the sample preparation area. Each sample is acidified with nitric acid and microwaved for 15 minutes, to remove any suspended particulate organic material.

ICP-MS Analysis

Prior to the installation of the first 7500i in October 2001, STL used a combination of ICP-MS, ICP-OES, Graphite Furnace AAS and Hydride generation for the elemental analysis of drinking waters. However, the limitations and time constraints of these techniques and the unreliability of their original ICP-MS led STL



Sample reception area at STL Bridgend - the samples in this photo represent 1.5 days of work.

Bridgend to evaluate a new system. Since the 7500i was brought on-line, productivity has improved significantly with approximately 300 samples per day being analysed throughout the working week. To date, STL have had no down-time with their first 7500i. Earlier this year, with the first 7500i running at full capacity, STL ordered a second 7500i. In order to help STL to quickly ramp up analytical capacity to meet the needs of new contracts, Agilent shipped and installed the new 7500i within just 2 weeks of receiving the order. STL are now transferring all metals analysis of drinking waters to the two 7500i systems while retaining their original ICP-MS system for lower priority work.

A Typical Day

To maximise sample throughput, data is acquired overnight and any data manipulation/report writing is carried out the following morning. The three principal ICP-MS operators, Kevin Denny, Stephen Lander and Roland Matthews, check the instruments and carry out any routine maintenance or cleaning, before loading the Cetac ASX-500 291-position autosampler, which includes 21 large vial positions for calibration and AQC samples, for acquisition of another overnight sample batch.

More information on STL and its services can be found at:
<http://www.stl-analytical.co.uk/>

Analysis of Organotin Compounds by GC-ICP-MS

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The presence of butyl and phenyl tin compounds in the environment has become more prevalent in recent years as a consequence of the wide use of plastic stabilizers, anti-fouling agents and use of biocides in agriculture. Once these compounds are released into the environment, they can undergo methylation and degradation processes to form new compounds that exhibit different bioavailability and toxicity properties. Because of the physicochemical properties (volatility, charge, polarity, molecular weight) of the resulting organotin compounds, speciation by GC may be preferable to LC, since GC provides better resolution and sensitivity¹. Also, introducing the sample in its gaseous state eliminates the need for desolvation/volatilization in the plasma which improves the robustness of the technique.

There has been keen interest recently in the coupling of GC to ICP-MS for low level elemental analysis of organometallic species in complex environmental samples. However, coupling a GC to an ICP-MS is technically challenging because of the risk of cold spots, active sites or dead volume in the transfer line between the GC and ICP-MS which could lead to chromatographic degradation. In addition, the effluent from the GC requires an additional carrier gas to achieve sufficient flow in the central channel of the plasma.

The Agilent GC-ICP-MS interface hardware consists of a torch adaptor and demountable GC torch with heated injector and a flexible heated transfer line. The GC column extends through the flexible transfer line and terminates within the ICP torch injector tube, just upstream of the plasma, so that separated species are carried directly into the plasma by a heated argon flow.

Species	L.O.D (ppt as Sn)*	%RSD** (at 0.1 ppb)	%RSD** (at 1 ppb)	R ² (to 50 ppb)
MBT (1)	2.5	18	9	0.9998
TPrT (2)	2.4	23	8	0.9995
DBT (3)	2.3	6	9	0.9993
MPhT (4)	4	13	13	1
TBT (5)	2.0	16	4	1
TeBT (6)	2.5	17	7	0.9999
TPeT (7)	2.2	13	7	0.9999
DPhT (8)	4.6	3	4	0.9966
TPhT (9)	5.5	10	12	0.9952

*3 sigma(n-1) blank/slope of the calibration curve (peak height)

**n=3 manual injections (peak area)

Table 1: Preliminary results showing Limits of Detection (LODs) of organotin species by GC-ICP-MS

Experimental

The preliminary results shown in Table 1 were obtained by coupling a 6890 GC and 7500 ICP-MS via the Agilent GC-ICP-MS Interface.

Addition of Optional Gases

In order to optimize the ICP-MS operating conditions, such as carrier gas flow and plasma forward power, xenon (m/z132) is used as the tuning mass. Xe may be present as an impurity in the Ar or can be added to the helium GC carrier gas. However the use of optional

gases, such as O₂ or N₂ added to the Ar carrier flow, has been shown to improve the performance of the GC-ICP-MS system. The addition of oxygen (5%) eliminates the formation of solvent-derived carbon deposits on the interface and improves sensitivity.

References

¹De la Calle-Guntiñas, M.B., Scerbo, R., Chiavarini, S., Quevauviller, Ph. And Morabito, R., Applied Organometallic Chemistry, **1997**, 11, 693.

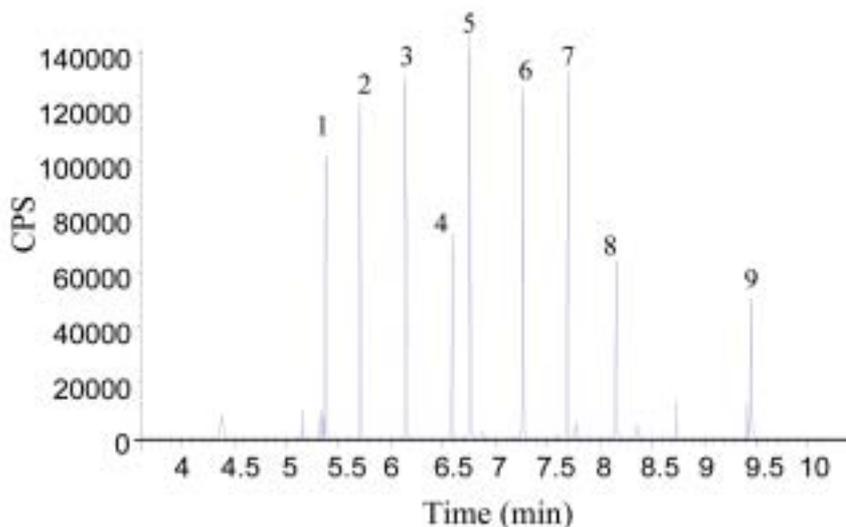


Figure 1: Optimum operating conditions for organotin species were found to be 5% oxygen and 1300W forward power. This chromatogram corresponds to a concentration of 10 ppb (as Sn) for each species.

Important - Download ICP-MS ChemStation Update Files

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Make sure you are getting the most from your 4500 or 7500 Series ChemStation software by updating to the latest software revision by downloading a patch file on-line. To check which Product number and Revision you are using, click on "Help"\ "About" in the ChemStation software. For example, anyone using the Agilent NT ICP-MS ChemStation (G1834A) revisions A.01.00-A.01.06 should update to

the latest revision A.01.07. Updating to the latest revision is important - it eliminates any minor faults that have been identified and fixed in earlier versions, and enables Agilent field staff to support you more efficiently.

A list of update and patch files available on-line is given in the Table 1. To download a file visit Agilent's "Software Status Bulletins and Patches" web site as follows:

1. Visit www.agilent.com/chem/icpms
2. Select "Technical Support" from the left hand panel
3. Select "Software Status Bulletins & Patches". (To access this page, you will need to enter the 10 digit registration number shown on the "Software Certificate and Registration Packet" supplied with your instrument)

4. Click on "ICP-MS ChemStation"
5. To download the relevant patch file for your ChemStation version, click on the relevant ChemStation Update and follow the instructions given.

If you experience any problems or have any questions, please contact your local Agilent Representative and request the patch file on a CD-ROM.

Instrument	ChemStation Product No.	Update from	Update to	Operating System	Patch File Name
7500	G1834B	Rev.B.01.00 - Rev.B.01.01	Rev. B.01.02	Windows 2000	Agilent 7500 ICP-MS ChemStation G1834B for Windows 2000 Patch File
7500 4500	G1834A	Rev.A.01.01 - Rev.A.01.06	Rev. A 01.07	Windows NT	Agilent 7500 / 4500 ICP-MS ChemStation G1834A for Windows NT Patch File
4500 without ISIS	G1821C Rev.C.01.01	Fixes minor faults, however Revision does not change		Windows NT	Agilent 4500 ICP-MS ChemStation G1821C Rev.C.01.01 and Supplemental Disk Rev.C.01.03 for Windows NT without ISIS Patch Files
4500 with ISIS	G1821C Rev.C.01.01	Fixes minor faults, however Revision does not change		Windows NT	Agilent 4500 ICP-MS ChemStation G1821C Rev.C.01.01 and Supplemental Disk Rev.C.01.03 for Windows NT with ISIS Patch Files
4500	G1821C Rev.C.01.00	Supplemental disk Rev.C.01.00	Supplemental disk Rev. C 01.01	Windows NT	Agilent 4500 ICP-MS ChemStation G1821C Rev.C.01.00 for Windows NT
4500	G1821B Rev.B.01.01	Supplemental disk Rev.B.01.02	Supplemental disk Rev. B.01.03	Windows 95	Agilent 4500 ICP-MS ChemStation G1821B Rev.B.01.01 for Windows 95

Table 1: 7500 and 4500 ICP-MS ChemStation patch files available on-line

Pre-assembled ISIS Tubing Kits

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The functionality of the 7500 Series ICP-MS can be enhanced via Agilent's Integrated Sample Introduction System (ISIS), which features high precision peristaltic pumps and optional switching valves to allow complete flexibility of configuration. ISIS offers the analyst an array of possibilities for sample introduction and can even be used for on-line sample preparation.

High Throughput

- Sample throughput can be greatly improved increasing instrument productivity. Normally sample uptake and rinseout rates are limited by the maximum flow of the nebulizer or the desire to minimize the instrument exposure to excessive sample flows. ISIS uncouples sample uptake and rinse flow from nebulizer flow, enabling the user to maximize uptake and rinse without

considering the limitations of nebulizer maximum flow. In this mode, the instrument is never exposed to high sample flows.

Auto-dilution

- Intelligent auto-dilution allows the analysis of "out of range", QC failure or high concentration analytes to be performed within a single automated run. Intelligent Sequencing software also required.

Discrete Sampling

- Discrete sampling allows the analysis of aggressive samples, % level dissolved solids and small sample volumes by analyzing a discrete sample volume, rather than a continuous sample stream.

The ISIS hardware is built into the 7500 chassis and the ISIS operating software is fully integrated into the

7500 ChemStation. ISIS is standard on the 7500i and available as an option on the 7500a, 7500s and 7500c models. An ISIS hardware and software upgrade is also available, giving users of any Agilent 4500 or 7500 model access to ISIS capabilities. Although setting up the tubing on the ISIS is not difficult, it can be time consuming, as each section of tubing has to be cut to the appropriate length and configured using a series of connectors. To simplify this process, Agilent has introduced pre-configured ISIS tubing kits as an option for those customers wanting to save time - each of the three types of pre-assembled kits can be connected easily in a matter of minutes.

	Part Numbers for:		
ISIS Tubing Kit Description	7500 Series ISIS Users	New 7500 Series ISIS Orders	New Agilent 7500i Orders
High Throughput	G3138-65023	G3148A #300	G3152A #300
Auto-dilution	G3138-65024	G3148A #301	G3152A #301
Discrete Sampling	G3138-65025	G3148A #302	G3152A #302

First European Plasma Prize



Agilent Technologies Europe has donated the **European Award for Plasma Spectrochemistry** to promote analytical plasma spectrochemical developments and applications in Europe. The prize will be awarded for either a single outstanding piece of work or for

continued important contributions to this field. Candidates must either be a European national or must have carried out the work in a European laboratory.

The prize will be awarded every second year and presented at the European Winter Conference on Plasma Spectrochemistry, where the winner will be invited to present a lecture, following the award ceremony. The prize, donated by Agilent Technologies, consists of EUR 5000 cash and an all-expenses paid trip to Japan to attend a scientific event or seminar, where the winner will also be invited to present a lecture. Travel expenses to the European Winter Conference to receive the award are also included.

Applications or nominations for the **2003 European Award for Plasma Spectrochemistry**, to be presented at the 2003 European Winter Conference on Plasma Spectrochemistry in

Garmisch-Partenkirchen, Germany, in January 2003, must be sent to the award panel chairman: Prof. Dr. Klaus Heumann (e-mail: heumann@mail.uni-mainz.de) by June 30, 2002.

More details regarding the award regulations can be found at the European Winter Plasma Conference website at:

<http://www.gdch.de/tagung/5545/index.htm>

New User Forum for ICP-MS Users

Agilent have introduced an ICP-MS User Community Forum which gives you, our users, the opportunity to post questions and responses on any aspect of your Agilent ICP-MS system. Users can learn and assist one another with method development, best practices or troubleshooting questions, and so on. Although Agilent will monitor the forum, the ICP-MS team will only become involved on a needs must basis. To get involved visit our web site and click on the "User Forum" link at:

www.agilent.com/chem/icpms

e-Seminars for ICP-MS Analysis

Speciation Measurement Using Hyphenated ICP-MS

Presenter: Ed McCurdy

Date: May 21st 2002

Time: 11:00 Central European Time

Language: English

Reaction Cell ICP-MS for Real World Applications

Presenter: Ed McCurdy

Date: May 21st 2002

Time: 15:00 Central European Time

Language: English

To register visit:

www.agilent.com/chem/icpms

and click on the relevant Highlight.

Up and Coming Events

Riva del Garda

May 13-17, 2002, Riva, Italy

40th Dutch Spectroscopy Group Meeting

May 23, 2002, Amsterdam, Netherlands

Laser Ablation ICP-MS Workshop

June 25 - 25, 2002

Utrecht, Netherlands

11th BNASS

July 8 - 10, 2002, Loughborough, UK

Agilent ICP-MS Journal Editor

Karen Morton

Agilent Technologies UK

This information is subject to change without notice

New Members of the Agilent ICP-MS Club

A very warm welcome to the following companies and institutions that have recently added an Agilent ICP-MS to their analytical facilities.

- Enviro Test Laboratories, Canada
- Public Works & Govt Services Req 61410, Canada
- Saskatoon Research Council, Canada
- Institute du Reserche Subatomique, France
- Chonbuk Uni, Korea
- Hantok Chemicals Co. Ltd, Korea
- Applied Chemie, Malaysia
- ABC Instrumentacion Analytica SA DE CV, Mexico
- Omegam, Netherlands
- Philips Lighting, Netherlands
- China National Environmental Monitoring Centre, PR China
- China National Import & Export, PR China
- Jilin Institute of Product Quality Supervision & Inspection, PR China
- Shenzhen CDC, PR China
- Xiamen EPA, PR China
- Antidoping Control Centre, Russia
- St Petersburg Water Research Center, Russia
- Inst. Hydrografica del Dero, Spain
- University of Granada, Spain
- Pharmaceutical Industry Technology, Taiwan
- Konya, Turkey
- Marmara U, Turkey
- University of Leeds, UK
- Rockwood Electronic Materials, UK
- Seven Trent Lab Bridgend 2, UK
- ABC Laboratories, USA
- ATMI, USA
- Lake Superior University, USA
- Commonwealth of Kentucky, USA
- Waste Management, USA
- Utah State University, USA
- Global Nuclear Fuels, USA
- Battelle, USA
- Friend Laboratory, USA
- Hi-Tek Environmental, USA
- Vietnam Nuclear Research, Vietnam

New Poster for Your Lab Wall

The first in a series of ICP-MS wall posters is now available. "Fundamentals of ICP-MS" explains the basic features of an ICP-MS and also some of the technology used in designing a modern ICP-MS instrument. The poster is designed to be printed in A1(594 x 841 mm, 23 3/8 x 33 1/8 inches), or A0 size (841 x 1189 mm, 33 1/3 x 46 13/16 inches), and is ideal for use in the lab or for teaching. Prints can also be encapsulated with a protective coating for use in the lab. To receive a copy of the poster in PowerPoint file format send a quick e-mail to karenmorton@agilent.fsnet.co.uk.

Alternatively visit the ICP-MS web site and download the pdf version.



Recent Agilent ICP-MS Publications

To view and download these latest publications, go to www.agilent.com/chem/icpms and look for "New Literature".

Title	Publication No.
Determination of Trace Metal Impurities in Semiconductor-Grade Hydrogen Peroxide	5988-5947EN
Determination of Trace Metal Impurities in Semiconductor-Grade Hydrofluoric Acid	5988-5421EN
Reliable, Real-time Analysis of Trace Metals on Silicon Wafer Surfaces (Tech Feature)	5988-5230EN
Designing Reaction Cell ICP-MS for Routine Use (Tech Feature)	5988-5612EN
Advances in Laser Ablation ICP-MS - Improving Dynamic Range and Data Acquisition Speed (Tech Feature)	5988-6198EN
New Wall Poster - Fundamentals of ICP-MS	5988-6486EN

