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**176Hf/177Hf Ratios: Resolution of 176Yb and 176Lu Interferences using ICP-QQQ**

Glenn Woods Agilent Technologies, UK

**Introduction**

Hafnium $^{176}\text{Hf}$ to $^{177}\text{Hf}$ isotope ratio analysis can provide insight into the different geological events and processes that a mineral underwent during its formation/metamorphosis. Hf has lower mobility than lead (Pb) in metamict minerals such as zircon, so it can offer an alternative to Pb/Pb or Pb/U dating in these minerals. Of the isotopes of interest, $^{177}\text{Hf}$ is relatively free from interference from other co-existing elements, but $^{176}\text{Hf}$ suffers isobaric overlap from $^{176}\text{Lu}$ and $^{176}\text{Yb}$.

The mass resolution required to separate $^{176}\text{Hf}$ from the Lu and Yb isobaric interferences ($M/\Delta M \approx 140,000$ for $^{176}\text{Lu}$ and $>150,000$ for $^{176}\text{Yb}$) is far beyond the capability of commercial High Resolution Sector Field ICP-MS (SF-ICP-MS), so sample preparation (chemical separation) is required prior to analysis. In cases where chemical separation cannot be performed, for example in-situ measurement by Laser Ablation (LA), the Lu/Yb overlaps mean that accurate Hf isotope ratio analysis is not possible.

An alternative ICP-MS approach is “chemical” resolution in a collision/reaction cell (CRC), using a specific ion-molecule reaction with a cell gas that either:

- Reacts with the interfering ion to neutralize it or move it to a new mass
- Reacts with the analyte to move it to a different, non-interfered mass

In this study, the second approach, known as “mass-shift”, was used. Hf reacts efficiently with ammonia to form Hf-ammonia cluster ions, while Lu and Yb are relatively unreactive. However, NH$_3$ cell gas reacts with many other ions, so control over the ions involved in the reaction process is essential, especially if isotopic information is to be preserved and potential new interfering species are to be eliminated. This requires a mass filtering step prior to the CRC, which is only possible with a tandem MS configuration. The Agilent 8800 ICP-QQQ has an additional quadrupole (Q1) positioned in front of the CRC, so is able to perform this analysis using its MS/MS capability. This method was used to measure Hf isotope ratios in a variety of samples containing Lu, Yb and mixed rare earth elements (REE).

**Experimental**

To test the ability of the Agilent 8800 ICP-QQQ to remove Lu and Yb isobaric interferences on $^{176}\text{Hf}$, a product ion scan of a Hf solution was performed with NH$_3$ in the CRC. Q1 was fixed at $m/z$ 176 and Q2 was scanned across the mass range to measure the Hf-ammonia reaction product ions (shown in Figure 1).

The most abundant ammonia adduct ion, $^{176}\text{Hf}$(NH)(NH$_3$)(NH$_2$)$_2^+$ occurs at $M+82$ amu ($m/z$ 258 for the $^{176}\text{Hf}$ isotope), and this adduct was selected as the preferred mass transition.

To check that the Hf isotope pattern was maintained in the product ions, a Neutral Gain Scan was performed where both Q1 and Q2 were scanned together, with a fixed mass difference of 82. Figure 2 shows the resultant spectrum, confirming that the Hf-ammonia product ions match the overlay of the theoretical Hf isotopic abundances.

Interferences were introduced from several sources to ensure no bias or new interferences had been created. The test solutions included:

- Hf standard (5 ppb) – also used for Mass Bias Calibration
- 100 ppb Yb & 5 ppb Hf
- 100 ppb Lu & 5 ppb Hf
- Mixed 100 ppb “REE1” standard and 5 ppb Hf
- Natural mineral sample containing approximately 500 ppm Ca, 120 ppm Mg, 15 ppm Na, 5 ppm K, 1500 ppm SO$_4$

**Figure 1.** Product Ion Scan from $m/z$ 170 to $m/z$ 260 showing ammonia adduct reaction product ions formed from $^{176}\text{Hf}$ precursor ion (Q1 set to $m/z$ 176)

**Figure 2.** Neutral Gain Scan of the Hf isotopes as ammonia clusters; theoretical Hf isotopic abundances are shown in red, confirming that the isotope ratios are preserved
The 8800 was set to measure $^{176/177}$Hf ratios using three different modes:

- No cell gas
- $\text{NH}_3$ reaction gas, Single Quad mode with Bandpass filter
- $\text{NH}_3$ reaction gas, MS/MS mode

Table 1 displays the Hf isotope ratio data for each of the test solutions in each instrument mode. It can be seen that there was a large positive deviation from the expected ratio in both Single Quadrupole modes of operation (no gas and ammonia with bandpass filtering). This indicates that “Single Quad” operation did not resolve the Yb and Lu isobars at m/z 176, or stop the formation of new reaction product ion interferences. By contrast, MS/MS mode with $\text{NH}_3$ cell gas gave consistent, accurate Hf IR data in all the sample matrices.

To visualise the reasons for the poor Hf isotope ratio performance in Single Quad mode, and to further investigate the potential overlaps, a mass scan of the mineral sample was performed using single quadrupole bandpass mode, with $\text{NH}_3$ reaction gas. The spectrum can be observed in Figure 3. The measured Hf isotopic pattern (far right of the spectrum) does not match the theoretical abundance template, showing that the Hf isotopes suffer overlap from new cell-formed cluster ions, due to the lack of control over the reaction processes.

As many of the matrix elements and other analytes can react with ammonia to produce higher order reaction products, MS/MS mode is essential to remove these precursor ions before they enter the cell and form new interferences.

Only the 8800 ICP-QQQ, which has a quadrupole mass filter operating at unit mass resolution positioned before the reaction cell, is able to control which ions enter the reaction cell. This ensures unprecedented levels of control over the reaction processes that occur in the cell. Any undesired side reactions are therefore eliminated and the underlying analyte isotope ratios are preserved.

**Conclusions**

The unique MS/MS capabilities of the Agilent 8800 ICP-QQQ were used to measure Hf isotope ratios with excellent accuracy – even in samples containing high levels of co-existing and potentially interfering matrix elements.

The isobaric overlaps from $^{176}$Lu and $^{175}$Yb on $^{176}$Hf were eliminated using $\text{NH}_3$ as the reaction gas. The reaction chemistry was controlled in the cell by operating the first quadrupole as a mass filter set to m/z 176. This excluded all ions apart from those at m/z 176 ($^{176}$Lu, $^{175}$Yb and $^{176}$Hf). Since only Hf reacted readily with $\text{NH}_3$, $^{176}$Hf was free to be measured via its most abundant cluster ion at m/z 258, which, together with the same ammonia cluster ion formed from the $^{177}$Hf isotope, allowed accurate Hf isotope analysis to be performed.

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**Table 1.** $^{176/177}$Hf isotope ratio (IR) data measured in samples containing various sources of interferences, using three different ICP-QQQ operating modes. The “deviation” is the error in the measured ratio relative to the true ratio of 0.282796.
Analysis of Undiluted Seawater using the Agilent 7900 ICP-MS with UHMI

Tetsushi Sakai and Ed McCurdy
Agilent Technologies

Seawater contains very high levels of salt matrix, mostly consisting of NaCl. The very low trace element levels found in uncontaminated, open-ocean seawater require special clean sample preparation and analysis facilities to enable accurate analysis. Near-shore seawater contains higher concentrations of trace elements, but routine analysis must still overcome the major obstacle of the tolerance of the ICP-MS to the high salt matrix. Coastal and estuarine waters may also contain high levels of organic matter and mineral elements, and salinity may vary significantly as a result of mixing with freshwater runoff.

Ultra High Matrix Introduction
Agilent’s patented UHMI technology increases ICP-MS matrix tolerance up to 25% total dissolved solids (TDS). UHMI also greatly increases plasma robustness, meaning that matrix suppression is reduced and good sensitivity is maintained for poorly-ionized elements.

Experimental Instrumentation
A standard Agilent 7900 ICP-MS fitted with the UHMI option was used for all measurements. Analyte isotopes, integration times and cell modes were defined by the selected Pre-set Method (EPA6020). Plasma conditions were defined by the Pre-set Method settings, and all other instrument tune settings were optimized automatically using the ICP-MS MassHunter autotune function. Instrument operating conditions are shown in Table 1. No manual optimization was required.

Table 1. ICP-MS parameters predefined in the "EPA6020" Pre-set Method

<table>
<thead>
<tr>
<th>Cell Mode</th>
<th>No gas</th>
<th>He</th>
</tr>
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<tbody>
<tr>
<td>Stabilization (sec)</td>
<td>0</td>
<td>5</td>
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<tr>
<td>Plasma Mode</td>
<td>HMI-8</td>
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<tr>
<td>RF power (W)</td>
<td>1600</td>
<td></td>
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<tr>
<td>Sampling depth (mm)</td>
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<td></td>
</tr>
<tr>
<td>Carrier gas (L/min)</td>
<td>0.69</td>
<td></td>
</tr>
<tr>
<td>Dilution gas (L/min)</td>
<td>0.28</td>
<td></td>
</tr>
<tr>
<td>Cell gas (mL/min)</td>
<td>Off</td>
<td>4.3</td>
</tr>
</tbody>
</table>

Table 2. Spike recoveries and stability from 8 hours analysis of undiluted CASS-4 and NASS-5. All elements were measured in He mode

<table>
<thead>
<tr>
<th>Element</th>
<th>Isotope</th>
<th>Recovery %</th>
<th>RSD%</th>
<th>Recovery %</th>
<th>RSD%</th>
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<tbody>
<tr>
<td>V</td>
<td>51</td>
<td>104.7</td>
<td>2.68</td>
<td>103.4</td>
<td>2.70</td>
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<tr>
<td>Cr</td>
<td>52</td>
<td>102.2</td>
<td>3.46</td>
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<td>Mn</td>
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<td>6.30</td>
<td>97.7</td>
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<tr>
<td>Co</td>
<td>59</td>
<td>96.6</td>
<td>1.85</td>
<td>96.7</td>
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<tr>
<td>Ni</td>
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<td>96.5</td>
<td>2.89</td>
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<td>2.92</td>
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<tr>
<td>Cu</td>
<td>63</td>
<td>104.4</td>
<td>2.83</td>
<td>103.1</td>
<td>2.97</td>
</tr>
<tr>
<td>Zn</td>
<td>66</td>
<td>100.8</td>
<td>16.74</td>
<td>96.1</td>
<td>10.86</td>
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<tr>
<td>As</td>
<td>75</td>
<td>106.4</td>
<td>2.74</td>
<td>106.4</td>
<td>2.14</td>
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<tr>
<td>Se</td>
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<td>95.5</td>
<td>9.76</td>
<td>95.7</td>
<td>7.84</td>
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<td>Mo</td>
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<td>1.60</td>
<td>99.3</td>
<td>1.44</td>
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<td>1.45</td>
<td>98.4</td>
<td>2.15</td>
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<tr>
<td>Sn</td>
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<td>102.8</td>
<td>2.28</td>
<td>102.8</td>
<td>2.76</td>
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<tr>
<td>Sb</td>
<td>123</td>
<td>113.1</td>
<td>2.07</td>
<td>112.7</td>
<td>2.20</td>
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<tr>
<td>Ba</td>
<td>135</td>
<td>94.9</td>
<td>5.67</td>
<td>94.6</td>
<td>5.41</td>
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<tr>
<td>Hg</td>
<td>202</td>
<td>112.5</td>
<td>4.72</td>
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<td>101.9</td>
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<td>103.8</td>
<td>1.71</td>
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<td>1.42</td>
<td>102.3</td>
<td>1.63</td>
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<td>Th</td>
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<td>108.8</td>
<td>1.96</td>
<td>108.9</td>
<td>2.21</td>
</tr>
<tr>
<td>U</td>
<td>238</td>
<td>106.4</td>
<td>2.07</td>
<td>106.1</td>
<td>2.69</td>
</tr>
</tbody>
</table>

Results and Discussion

Spike recovery and stability
Approximately 140 samples of the seawater certified reference materials (CRMs) CASS-4 and NASS-5 were analyzed undiluted, during a sequence lasting ~8 hours. The samples were run alternately unspiked and with a spike containing the analytes at 10 µg/L (Hg at 1 µg/L). Trace elements were quantified against a simple aqueous calibration, and the spike recovery results are shown in Table 2.

Excellent spike recoveries were achieved, with most elements within +/- 5% of the spiked value, and with RSD <5% for the entire 8 hour sequence. This is a remarkable result, considering that the analytes were measured in a high salt matrix against non-matrix-matched calibration standards. The good recovery of the poorly ionized elements Zn, As, Se, and Cd, demonstrates the ability of UHMI to reduce ionization suppression.

The internal standard signals were monitored throughout the sequence, and the ISTD recoveries are shown in Figure 1. The good stability of the ISTD signals confirms that the 7900 ICP-MS with UHMI is suitable for routine analysis of undiluted seawater samples, with very little signal loss throughout the 8 hour sequence, and consistent ISTD signals for low and high mass elements, and those with low and high ionization potentials (IPs). This consistency makes internal standard correction and in-run quality control much simpler and more reliable.

Conclusions
The Agilent 7900 ICP-MS with UHMI tolerates high and variable TDS levels, allowing stable, accurate, long-term analysis of high matrix samples such as seawater, without needing any dilution prior to analysis.

Figure 1. Internal standard signal recoveries in 140 undiluted seawater samples over~ 8 hours. Recovery limits (dotted red lines) are at 80% and 120%.
China Hosts First Agilent-sponsored Petroleum R&D Conference

Wayne Collins Agilent Technologies

Agilent sponsored the first petroleum R&D conference in Beijing in May 2015 that was attended by over 200 researchers. The event featured four speakers from the Department of Earth & Atmospheric Sciences at the University of Houston, as well as scientists from Chinese universities and corporations. The program focused on the application of advanced instrumentation, primarily mass spectroscopy based, for the characterization of crude oil.

Professor Adry Bissada, Director of the Petroleum Systems and Geochemistry Program, set the stage for the program with his presentation on using the characteristics of crude oil to deduce the characteristics of the source rock—a process that he termed geochemical inversion. Subsequent presentations illustrated how this concept is applied through two different approaches—one focused on the inorganic constituents of the oil and the other on the organic components.

Dr. John Casey, Director of The Inductively Coupled Plasma Research Laboratory, described a process for determining up to 47 elements in crude oil at ppb levels and using these data as a “fingerprint” related to the source. This study utilized both ICP-OES and ICP-MS to examine crude samples from different regions of the world. Each sample produced a different pattern and inferences could be drawn as to the geological source and conditions of its formation. Data from 20 wells from a single field in California are displayed as a log-scale “radar” plot in Figure 1. In addition to providing data meaningful in petroleum exploration and reservoir characterization, this technique can also be applied in identifying the source of petroleum released into the environment. For example, the elemental map obtained for “tar balls” found on the beaches of Southern California can be compared to the elemental map for potential sources such as seabed seepage sites or drilling operations off the coast.

Dr. Yongjun Gao, Research Professor at the University of Houston, described a procedure for the precise determination of vanadium isotope ratios in crude oil. V isotope ratios complement the vanadium/nickel ratios and abundances commonly used as a geochemical tracer for depositional environment of the source rock, and index of relative thermal maturity and oil-oil and oil/source rock correlations. The procedure involved microwave digestion of the crude oil, followed by removal of interfering ions by treatment with both cation and anion exchange columns. The prepared solution can then be analyzed for $^{50}\text{V}$ and $^{51}\text{V}$. The mechanisms that control the V isotope fractionation in crude oils will be further investigated with the ICP-QQQ and LC-ICP-QQQ by correlating V isotope composition with other elemental concentrations, V molecular speciation and pyrolysis experiments. ICP-QQQ is also essential in evaluations of eluted fractions to establish complete recovery of V.

Presentations using organic MS included “Using GC/MS to explore oil foundation principle and oil bio marker analysis” by Dr. Zhang Zhirong, Chief Researcher from Wuxi Research Institute of Petroleum Geology at the Sinopec Research Institute of Petroleum Exploration and Development; “Applications of GC-QQQ and GC-IRMS in unraveling origin and thermal maturity of condensates and associated natural gas” by Ms. Mei Mei, Doctoral Candidate at the University of Houston; “GC-QTOF for low-content sulfide analysis in diesel” by Mr. Liu Zelong, Professorate Senior Engineer at the Sinopec Research Institute of Petroleum Processing; “Newest characterization technology for complex petroleum components: Ion mobility Q-TOF LC/MS” by Professor Shi Quan, Deputy Head of the State Key Laboratory of Heavy Oil Processing at the China University of Petroleum (Beijing); and “GC-MS technique application in olefin catalyst research and quality control” by Ms. Zhang Ying, Professorate Senior Engineer at the Sinopec Beijing Research Institute of Chemical Industry.
Collaboration Kicks Off with Opening of ICP-MS Laboratory at the NPL Radioactivity Group

Ben Russell, Richard Brown
NPL, London, UK
Raimund Wahlen Agilent Technologies

An Agilent 8800 Triple Quadrupole ICP-MS (ICP-QQQ) was recently installed in a newly configured lab at the Radioactivity Group of the National Physical Laboratory (NPL), London, UK. The laboratory was fitted with a new air handling system and fume cupboard to minimize the risk of cross-contamination of samples. The facility will deliver novel radionuclide measurements, addressing the needs in nuclear decommissioning, environmental radioactivity monitoring, radioactive decay metrology and nuclear forensics. Novel methods will be developed to allow traceability to the Becquerel for the analysis of difficult to measure radionuclides in complex matrices, without the need for extensive sample preparation. Traditional radiochemical techniques alone are unlikely to provide sufficient accuracy and speed, and must be augmented by innovative ICP-MS based techniques.

Extending The Range of Nuclear Applications
The 8800 ICP-QQQ operates in MS/MS mode, which can be used for removal of isobaric interferences (e.g. 90Zr on 90Sr, or 135Ba on 135Cs), extending the range of nuclides that can be measured and improving the accuracy of radionuclide analysis. This opens up the possibility of rapid measurement of radionuclides in complex samples, without the need for extensive chemical separation prior to sample introduction.

Ultimately, the techniques developed at NPL will therefore offer significant economic benefits to nuclear sites.

The research at NPL will address the increasing demand for accurate low-level measurement of medium and long-lived radionuclides such as 90Sr, 90Zr, 151Sm, 135Cs and 129I, and a reduction in the measurement uncertainty in samples relevant to the nuclear industry. The aim is to develop robust, sensitive procedures for a range of radionuclides and sample matrices that will be adopted by the nuclear industry and the associated supply chain.

Additional applications of interest include the measurement of nuclear fuel activation products including 236,237Np and 239,240Pu; tracer elements such as 236Np and 244Pu; and isotope ratio measurements relating to nuclear forensics, including 135Cs/137Cs and 238U/238U.

Internal and quality control standards will be developed, which may also be used as reference materials – an area that has been identified as a limitation in previous studies of radionuclides by ICP-MS. The instrument will also be used in combination with the radioanalytical expertise at NPL for standardization of radioisotopes to provide accurate half-life measurements.

Information Sharing
As well as publication in high impact journals, the new ICP-MS applications will be developed into best-practice guides and documentary standards that can be transferred to other laboratories across the industry.

Second ICP-QQQ on Order
Since the opening of the new ICP-MS facility, NPL’s Environment Division has purchased an Agilent 8800 ICP-QQQ. The second instrument will be used primarily for air monitoring measurements.

A Collaborative Approach
Representatives from NPL and Agilent celebrated the opening of the ICP-MS lab (pictured). Going forward, both companies will work closely on method development for nuclear applications using ICP-MS. The facility will also be used for training purposes.

Cake in the shape of an Agilent 8800 ICP-QQQ, to celebrate the opening of the new lab

Agilent works closely with other UK-based institutions, including LGC and the Geoscience Advisory Unit, Southampton. Both of these facilities also use Agilent 8800 ICP-QQQ.

For further information on NPL, contact Ben Russell: ben.russell@npl.co.uk
Software Tips and Tricks

ICP-MS MassHunter 4.2: Uploading Batch Results to LIMS

Steve Wilbur
Agilent ICP-MS Software Product Manager

Connect
ICP-MS MassHunter 4.2 (revision C.01.02) includes a powerful, user configurable tool for compiling batch results for upload to a Laboratory Information Management System (LIMS). Configuring selected sample and analyte information for a batch is as easy as clicking on the desired information in the LIMS Configuration Tool, specifying the output file format (.csv or .txt) and entering the desired name and location for the output file.

Configure
The LIMS Configuration Tool is accessed from Reports under the Settings Gadget in the Data Analysis window.

Clicking Configuration under LIMS Settings will open the LIMS Configuration pane. From here, select either Sample or Analyte from the Data type list box (upper left corner). The available data for the selected Data type will be displayed. Simply double clicking the desired information (or clicking and selecting “Add ->“) will move the data selection to the Output Data list box for inclusion in the LIMS output file. After specifying which data are to be included in the Output file, the order in which the data items appear can be changed by using the Move Up or Move Down buttons.

Additional formatting information is easily configured via the check boxes down the left side of the pane. The output File Path, File Name and .csv or .txt Extension can be user-specified, or, if no location or filename is selected, the LIMS output file will automatically be given the same name as the Batch, and placed in the /agilent/ICPMS/LIMS folder.

Execute
After the LIMS file format configuration is saved, sending batch results to LIMS is as simple as selecting the Reports Gadget in Data Analysis and clicking LIMS – Export Selected Samples.

Done!
The creation of the LIMS output file is fast, though the actual speed will depend on the batch size, the PC configuration and other processes which may be running. On our test system, uploading a batch containing 100 samples, each with more than 50 analytes, took less than 30 seconds. Once the file creation and upload has been completed, a confirmation message is displayed.

More Information
agilent.com/chem/icpmsmasshunter
You Are Invited: Agilent Events at Winter Plasma Conference 2016

Join us at the 2016 Winter Plasma Conference, January 11-16, 2016 in Tucson, Arizona, USA. We will be holding several customer events including lunch seminars on our latest technology, and a special customer evening event.

For more information on the conference, please visit: http://icpinformation.org For more information on Agilent activities at the conference, please contact your local Agilent ICP-MS Product Specialist.

Conferences. Meetings. Seminars.


Webinar: Fingerprinting of Crude Oil using Triple Quadrupole ICP-MS and ICP-OES

Learn more about “Fingerprinting of Up to 57 Trace and Minor Elements in Crude Oil Using a Single Sample Preparation Method” in this informative webinar presented by Dr. John F. Casey, Professor of Geology, Department of Earth & Atmospheric Sciences, University of Houston.

First hosted by C&EN on 20 Oct 2015. Catch up now from: http://cen.acs.org/media/webinar.html

Agilent ICP-MS Publications

To view and download the latest ICP-MS literature, go to www.agilent.com/chem/icpms

- Application note: Determination of pesticides in foods using phosphorus and sulfur detection by GC-ICP-QQQ, 5991-6260EN

- Application note: Routine, high-throughput, multi-element analysis of milk and milk powder using the Agilent 7900 ICP-MS, 5991-6185EN

- Application note: The impact of vineyard origin and winery on the elemental profile of red wines, 5991-6111EN

- Application note: Fast and accurate absolute-quantification of proteins and antibodies using Isotope Dilution-Triple Quadrupole ICP-MS, 5991-6118EN

- Application note: Determination of trace elements in steel using the Agilent 7900 ICP-MS, 5991-6116EN

- Application note: Quantitative analysis of high purity metals using laser ablation coupled to an Agilent 7900 ICP-MS, 5991-6156EN

This information is subject to change without notice.

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