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The Measure of Confidence



Agilent Technologies

Introducing the Agilent/Varian Range of Atomic Spectrometry Instruments

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Agilent Technologies' US\$1.5 billion acquisition of Varian was finalized in May 2010. This acquisition, the largest in Agilent's history, adds a new range of Atomic Spectroscopy instruments to Agilent's portfolio, which are profiled here.

Agilent Technologies is well known as the leading provider of GC, GC/MS, LC and LC/MS products, but has also achieved unprecedented success with the 4500 Series, 7500 Series and 7700 Series ICP-MS instruments. Agilent entered the worldwide ICP-MS market in 1994 with the launch of the then revolutionary 4500 Series ICP-MS, and until the acquisition of Varian, Agilent's ICP-MS products were the only inorganic analytical instruments offered by the company. With the addition of Varian's world-renowned AAS and ICP-OES products, Agilent becomes an even greater resource and partner for our customers.

Agilent is now able to offer the full range of atomic spectrometry instruments:

- AAS (Atomic Absorption Spectroscopy) with flame, vapor generation, graphite furnace or a combination of these techniques
- ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy)
- ICP-MS (Inductively-Coupled Plasma-Mass Spectrometry)

The analytical capabilities that have made the 7700 Series ICP-MS such an outstanding success since its launch in 2009 are further complemented by the addition of Varian's range of AAS and ICP-OES products. This enhanced portfolio provides Agilent customers with an even wider range of options to suit the diverse analytical requirements of many applications and industries.

Flame Atomic Absorption Spectroscopy



Varian AA240 FS AAS

Flame AAS is typically used for the determination of a small number of elements at relatively high concentrations (low ppm to % level), often in very high matrix samples. FAAS is easy to use, setup and maintain, and is relatively inexpensive to buy and run.

Varian's range of Flame AAS instruments includes the AA240 FS and AA280 FS which feature patented "Fast Sequential" (FS) capability. This allows multiple elements to be measured sequentially in each sample, dramatically speeding up sample throughput in applications where the number of analytes to be determined is not high enough to justify purchase of an ICP-OES.

Graphite-Furnace Atomic Absorption Spectroscopy

Graphite Furnace AAS is a single element technique designed for the determination of elements at very low detection limits (low ppb or ppt). GFAAS is particularly suitable when

only a small number of analytes are required, or when sample volumes are extremely small. Varian's GTA120 graphite furnace atomizer can be added as an accessory to any Varian Flame AAS system, to give dual or switchable flame/furnace operation.

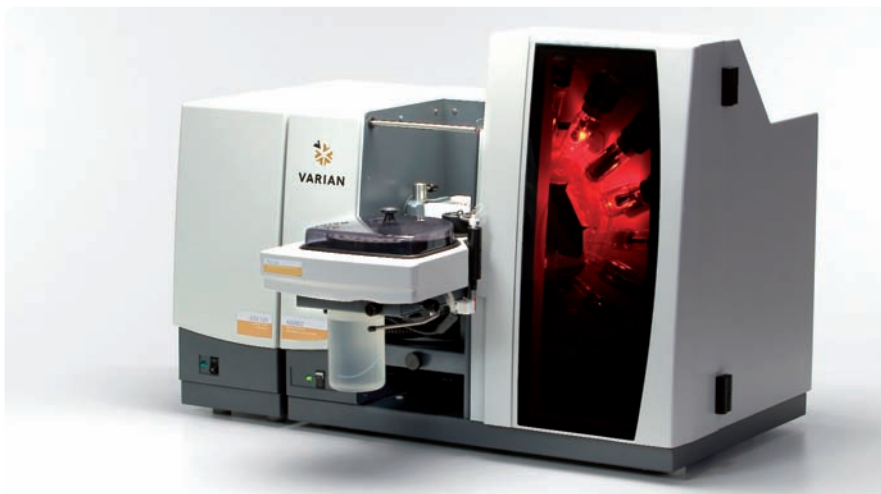
In addition, Varian's GFAAS range includes the AA240 and AA280 Zeeman systems which are dedicated GFAAS instruments with Varian's unique transverse, modulated Zeeman background correction technique. Zeeman background correction was patented by Varian in the 1970s and remains the preferred correction technique for regulated environmental analysis using GFAAS.

Inductively Coupled Plasma Optical Emission Spectroscopy

ICP-OES, also known as ICP-Atomic Emission Spectroscopy (ICP-AES), uses an argon plasma excitation source which is fundamentally the same as the plasma used in ICP-MS. However, an ICP-OES measures the optical emission from excited analyte atoms and ions in the plasma, whereas an ICP-MS extracts the ions produced in the plasma and passes them into a vacuum system to be separated and measured directly using ion counting.

ICP-OES systems are available in two common configurations: Radial ICP, where the plasma is viewed from the side, provides the best tolerance to dissolved solids and organic solvents. Axial ICP, where the plasma is viewed end-on, provides the best sensitivity, due to the longer viewing region and the absence of the intense emission from the side of the plasma.

ICP-OES instruments cover many of the same applications addressed by



Varian AA280 Zeeman AAS including the GTA 120 Zeeman Graphite Furnace Atomizer



Varian 700-ES Series ICP-OES

ICP-MS, except that typical ICP-OES detection limits (DLs) are in the single- to sub-ppb range, compared to single- to sub-ppt DLs for ICP-MS. However, both sample throughput and matrix tolerance are better on ICP-OES, because the optical spectrometer does not require a vacuum interface and matrix deposition is therefore eliminated.

Developments in the technology of both ICP-OES and ICP-MS have led to greater overlap between the two techniques. As a result, the choice of which technique is best for a given laboratory and application is often dictated by practical factors such as the available budget, the list of analytes, the required reporting limits, and the methodology and expertise already established in the laboratory.

In many respects, ICP-OES and ICP-MS are complementary techniques, with ICP-OES being ideally suited to the measurement of higher analyte levels in very high matrix samples, and ICP-MS delivering much lower DLs in lower matrix samples. Many busy laboratories can justify the presence of both techniques for different applications. ICP-MS does offer an additional capability, as it delivers low DLs for elements that are difficult to measure directly with ICP-OES, such as As, Se, Sb and Hg.

The Varian 700-ES range of ICP-OES instruments includes the 710/715-ES, 720/725-ES and 730/735-ES, covering both dedicated axial and radial configurations. The 710/715-ES is an entry level ICP-OES which is ideal

for laboratories analyzing low to medium numbers of samples. Both the 720-ES and 730-ES Series feature a patented CCD (charge coupled device) detector that delivers the world's most flexible, best performing and fastest ICP-OES platform. The 730-ES Series has been configured for maximum productivity, and includes a fully automated switching valve, 4-channel pump and mass flow control of the nebulizer gas.

Inductively Coupled Plasma Mass Spectrometry

Completing the line-up of inorganic or atomic spectrometry products is the Agilent 7700 Series ICP-MS. Launched in 2009 as the replacement for the highly successful 7500 Series, the 7700 Series has rapidly eclipsed the previous model, being accepted as the new standard for high performance high-throughput ICP-MS.



Agilent 7700 Series ICP-MS

The 7700x model includes the patented HMI (High Matrix Introduction) capability, which provides matrix tolerance up to 10x higher than conventional ICP-MS systems, allowing routine analysis of samples containing up to 1 or 2% total dissolved solids. Also standard on all 7700 Series instruments are the 3rd generation Octopole Reaction System (ORS³) operating as standard in helium (He) mode, and the true 9 orders dynamic range detector. Together these provide unparalleled interference removal, analytical range and simplicity of operation.

Looking Ahead

The 7700 Series has redefined the capability of ICP-MS, and Agilent's market-leading ICP-MS systems are now joined by innovative and powerful AAS and ICP-OES products from Varian. The combined product range provides our customers with new opportunities as we enter an exciting new chapter in Agilent's history.

Learn More

More information on Agilent's atomic spectrometry products can be found at:

www.agilent.com/chem/icpms and www.varianinc.com/products/spectr/index.html

New 7700e ICP-MS and MassHunter Software Revision for the Agilent 7700 Series ICP-MS

Ed McCurdy ICP-MS Product Marketing, Agilent Technologies

A new revision of ICP-MS MassHunter software has been released for the 7700 Series, providing many ease-of-use, functionality and productivity benefits for 7700 Series users.

The new revision of ICP-MS MassHunter is standard with all new 7700 Series instruments from October 2010 and is also available as an upgrade for existing 7700 Series users. The new revision provides a new interface for instrument control, method setup and sequencing. It is focused on ease of use for routine operations, and is configured in two different versions:

- “Core” version, which is supplied as standard with the newly-released 7700e ICP-MS
- “Full” version, which is supplied with the 7700x and 7700s models.

The 7700e is a new addition to the 7700 Series line-up, providing a cost-effective solution for many routine applications where a high degree of automation is required for a routine application. The 7700e with Core version of MassHunter provides a simplified and streamlined software interface, together with a standardized hardware configuration. Upgrades to 7700x specification hardware and software are available, if future lab needs dictate that more flexibility is needed for advanced applications.

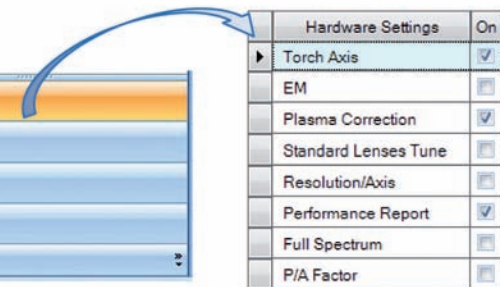


Figure 1. Startup

MassHunter Key Features

The latest revision of MassHunter software for the 7700 Series ICP-MS is designed around a new graphical user interface, where all the critical information required at each stage of system operation is clearly presented in a single window. In addition to the existing Data Analysis module, the key panes of the new software are:

- **Startup.** Allows the user to pre-define a series of standard optimization tasks and performance checks to be run each time the plasma is ignited.
- **Hardware.** Provides a single, integrated window for setup and control of hardware functions.
- **Batch.** The batch pane contains all the information required for each experiment or sample analysis job.
- **Queue.** The run control window, providing a real-time display of batch status and current sample acquisition.

The main features of each of these panes are outlined below.

Startup

The Startup function in the new MassHunter software provides a user-configurable list of hardware optimization tasks and checks that can be run automatically each time the plasma is ignited. This simple and automated approach ensures that all hardware systems are operating at their optimum, and gives a continuous record of system performance. Startup is particularly useful in busy commercial laboratories, as it ensures that performance is consistent from day to day, and is independent of the operator’s expertise.

In the example illustrated in Figure 1, Startup is configured for the hardware optimization tasks that might be run

Figure 2. Hardware

following routine maintenance of the sample introduction. Torch Axis (horizontal and vertical alignment with the interface orifice) and Plasma Correction are followed by the generation of a standard Performance Report, acquired with pre-defined warm-up delay for consistency.

Hardware

The Hardware pane contains a graphic of the main system components, providing the user interface for all Hardware functions, including system configuration, individual sub-system optimization and maintenance, early maintenance feedback (EMF), diagnostics, and Performance Reports (shown in Figure 2). Using a single user interface for all system setup, maintenance and diagnostic functions ensures that users have all the information on system operation easily available at all times.

The Performance Report provides a graphical display of key system performance parameters (sensitivity, oxides, background, mass calibration and resolution, etc). The report viewer also includes a History view, which allows users to monitor system performance over time. In addition to system performance, the Performance Report also saves all the current tune and meter values, giving a record of system operating parameter settings.

The instrument graphic updates automatically when different system hardware modules (such as a different autosampler or LC/GC modules) are selected.

Batch

The Batch pane brings together all the information required for a sample analysis session or experiment, including peripump program, tune modes, acquisition parameters (illustrated in Figure 3), sample list and data analysis (calibration) parameters. All these parameters are saved in the Batch and can be

recalled for use in subsequent sample analysis.

The Batch concept is a major advance for the ICP-MS MassHunter platform, as it simplifies file management and ensures that consistent conditions can be used for regular or routine sample analysis. A new Batch can be created from a blank template, an existing Batch, or one of a series of Pre-set Methods which ship with the software. Pre-set Methods are a great innovation in MassHunter, as they pre-define all critical parameters for many typical applications.

A unique feature of Pre-set Methods on the 7700 is the use of Pre-set Plasma Conditions, where settings suitable for low, medium or high-

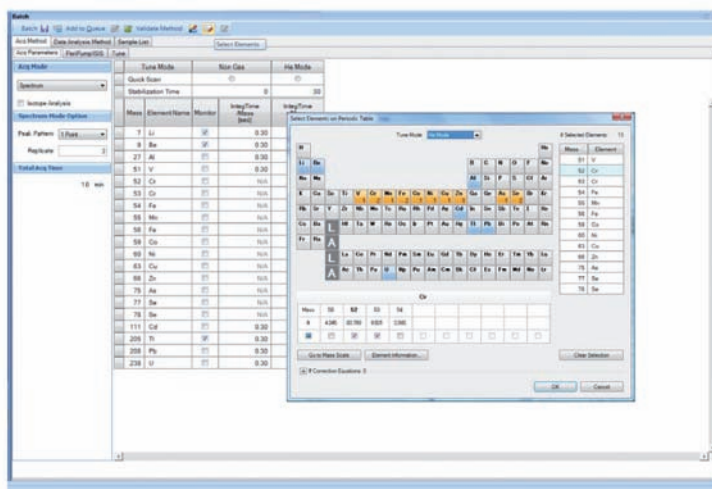


Figure 3. Acquisition parameters

At any time during sequence analysis, the current sample list can be edited,

Data Analysis

The previous version of ICP-MS MassHunter software introduced the databatch table, and this layout is retained in the new version (shown in Figure 5). However, the Data Analysis method is now integrated in the Batch, so the calibration table information is retained when a routine sample analysis is repeated.

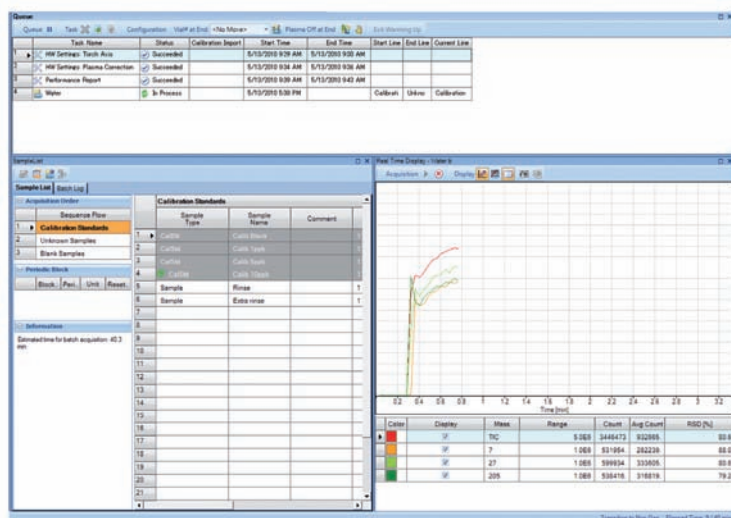


Figure 4. Queue and Signal Monitor

matrix samples can be preselected and used repeatedly, based on a “plasma map” which is automatically calibrated for each instrument.

for example to add a rush sample, and scheduled tasks and Batches waiting in the Queue list can also be moved, edited or deleted.

Another new function added in the Data Analysis module of the latest revision of MassHunter is the facility to process Quick Scan data. Quick Scan is a new addition for MassHunter and allows users to include a fast, full mass spectrum acquisition for each sample in the batch, giving access to semiquant or screening data for all elements in all samples. This is a very useful function in cases where the sample composition is unknown, and where there may be interest in elements not specifically calibrated and measured quantitatively in the method.

Queue

The Queue contains an interactive schedule listing individual Hardware tasks (such as the performance checks run during Startup) and Batches awaiting analysis. As with all panes, the Queue screen is user-configurable, but the default layout (shown in Figure 4) includes the Queue list, the interactive sample list for the current batch, and the real time display for the current sample.

Figure 4 also illustrates the new Monitor function, which displays the signals for several user-selected masses in the period between acquisitions. As well as a visual rinse monitor, this provides an indication that the next sample has reached a steady signal before acquisition.

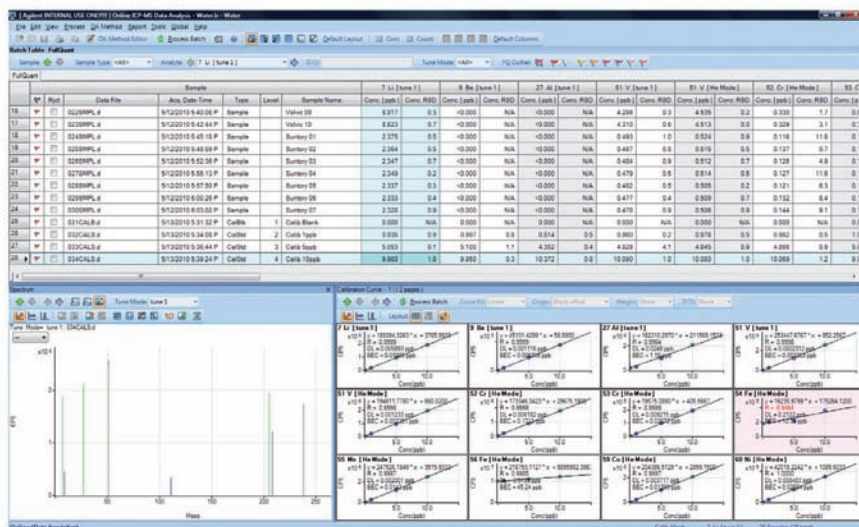


Figure 5. Data Analysis Batch view

Determination of Brominated Flame Retardants in Plastic by HPLC-ICP-MS

John Entwisle and Heidi Goenaga-Infante

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Introduction

Brominated flame retardants (BFRs) are a major group of flame retardants, which have been added to plastic/polymeric materials such as electrical equipment, textiles, automobile trim, etc. BFRs were merchandized as technical mixtures (penta, octa and deca) with increasing degrees of bromination.

There are two major families of interest, namely polybrominated biphenyls (PBBs) and polybrominated diphenyl ethers (PBDEs). Chemically, PBDEs are aromatic compounds substituted with up to 10 bromine atoms on the core diphenyl ether molecule. PBBs are the bromine homologues of PCBs.

Increasing concerns over neurotoxicity and endocrinal disruption have led to the inclusion of PBBs and PBDE's in the Restriction of Hazardous Substances (RoHS) Directive (2002/95/EC) and the Waste Electronic and Electrical Equipment (WEEE) Directive (2002/96/EC). In the European Union, Commission Decision 2005/618/EC specifies a limit of 1 g/kg (0.1%) for the sum of PBDEs and PBBs in plastics [1].

There are a number of significant analytical challenges associated with the accurate determination of BFR in plastics [2]. They include the possible co-extraction of compounds that may interfere in the analysis, the difficulty of achieving efficient extraction while preserving compound integrity, particularly as heat and UV light can cause BFR breakdown.

The results of a recent international intercomparison (CCQM-P114) organized to evaluate the state-of-the-art measurement capabilities for selected PBDEs in plastic [3] highlight that although GC-MS has been the most popular method for BFR analysis, a degree of de-bromination can occur during the gas phase separation.

Therefore liquid chromatography could provide a suitable alternative as the separation is performed at ambient temperature thus minimizing possible thermal de-bromination. Figure 1 shows the mechanism by which PBDE-209 undergoes de-bromination leading to formation of PBDE-206.

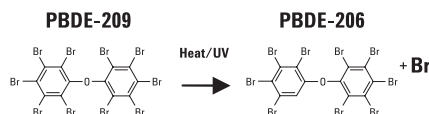


Figure 1. Formation of PBDE-206 by de-bromination of PBDE-209

In this work, a straightforward accelerated solvent extraction (ASE) method was used to extract selected BFRs (PBDEs 47, 183, 206 and 209 and PBB-209) from plastic. Toluene was used as the extraction solvent, and the BFRs were separated using HPLC-ICP-MS and quantified by external calibration. The method was validated by analysis of the European Reference Material: ERM-EC591.

Experimental

Sample Preparation

ERM-EC591 (polypropylene pellets) was cryogenically ground through a 0.5 mm mesh using a centrifugal mill (Type 17-140 Glen Creston Ltd, London, UK). The material was stored in an amber vial at -20°C. Compound extraction was performed using accelerated solvent extraction employing an AS200 (Dionex Ltd, Camberley, UK) at a temperature of 90°C. Approximately 0.1 g of sample was accurately weighed into a 1cm³ cell. Ottawa sand (Fisher Scientific Ltd, Loughborough, UK) was used as a dispersing agent and cellulose filters placed at either end of the cell. Toluene (Fisher Scientific Ltd, Loughborough, UK) was used as the extraction solvent. The ASE extracts were also accurately weighed.

Reagents and standards

Single 50 ug/mL congener standard solutions of PBDEs 47, 183, 206 and 209 and PBB-209 were purchased from LGC Standards Ltd, Teddington, UK. Standards and extracts of ERM-EC591 were diluted (1:1) by weight with the internal standard solution (in house purified 9-Bromoanthracene at 40 mg/kg) before analysis.

HPLC-ICP-MS Analysis

The HPLC-ICP-MS system consisted of an Agilent 1100 HPLC coupled to an Agilent 7500ce ICP-MS. The ICP-MS operating conditions are summarized in Table 1. Separation of the selected congeners was achieved on a Phenomenex (Macclesfield, UK) Luna phenyl-hexyl column, (3 µm, 2 mm i.d, 100 mm length) using a methanol concentration gradient [82 to 93 % (v/v) MeOH over 60 minutes] at a flow rate of 0.18 mL/min. To minimize the problems caused by introducing high amounts of methanol into the ICP (e.g. deposition of carbon in the cones, plasma instability, etc), the spray chamber was maintained at -5°C, the RF power was increased and oxygen was added as the optional gas (see Table 1). BFR quantification was performed by external calibration using the combined peak areas (m/z 79+81) with the peak from 9-bromoanthracene providing internal standard correction.

Nebulizer	Agilent MFN
Cones	Pt sampler, Pt skimmer
RF Power (W)	1540
Carrier Ar gas (L/min)	0.66
Makeup Ar gas (L/min)	0.19
Optional plasma gas flow	Oxygen (100 mL/min) valve set 5%
Spray chamber temp (°C)	-5
Acquisition parameters	
m/z	⁷⁹ Br; ⁸¹ Br
Int. time per point (sec)	0.75
Total acq. time (min)	60

Table 1. ICP-MS operating conditions

Results and Discussion

Total Br Content of ERM-EC591

Using PBDE-47 (with purity traceable to LGC) as a calibrant and utilizing the peak area summation technique over the entire chromatogram, the total Br content of the extract was calculated. An average recovery of 102% with an RSD of 2.5% (n=6) for total Br from the certified value (2.08 g/kg with an expanded uncertainty (k=2) of 0.07 g/kg) was achieved.

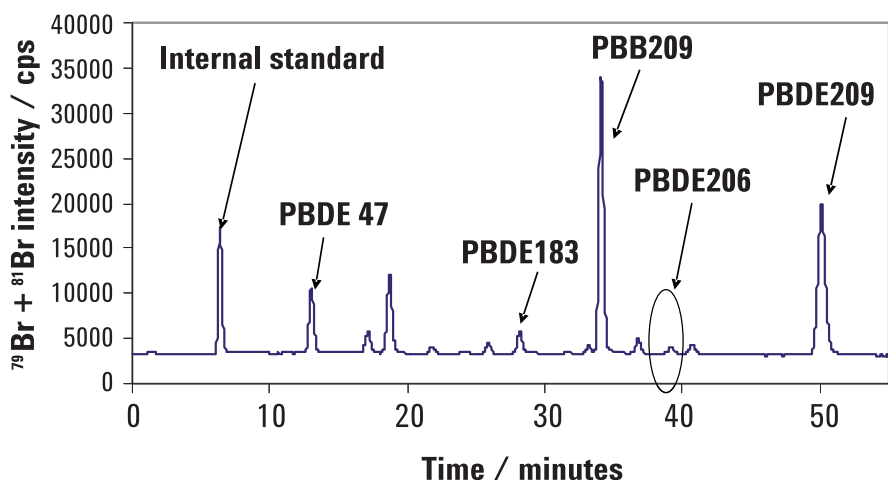


Figure 2. HPLC-ICP-MS chromatogram of an extract of ERM-EC591

Quantification of BFR in ERM-EC591 by HPLC-ICP-MS

Using the methodology described here, the quantitative analysis of PBDEs 47, 183, 206 and 209 and PBB-209 in the ERM material was undertaken. Figure 2 shows an HPLC-ICP-MS chromatogram of an ERM-EC591 extract. The average recoveries obtained for PBDEs 47, 183 & 209, and PBB-209 ranged from 93.6% to 105.6%, as shown in Table 2. It is interesting to note that the recovery of PBDE-206 is relatively poor. In the absence of a certified value for this compound, the recovery has been calculated on the basis of a reference value obtained by GC-based techniques during the certification of this material. The reference value was calculated on the basis of only four data sets and the associated uncertainty contribution of the spread of laboratory mean values was 18% [3]. As noted earlier, de-bromination of PBDE-209 with formation of PBDE-206 has been shown to occur during GC separation of BFRs. This could be a possible explanation for

the difference observed between the reference value published [3] and the mass fraction obtained for PBDE-206 by the LC-based methodology described here.

Conclusions

The methodology developed and validated here, which is based on the combination of accelerated solvent extraction followed by quantification using HPLC with quadrupole ICP-MS and external calibration is a promising and straightforward tool for the accurate determination of selected PBDEs and PBBs at tens to hundreds mg/kg levels in plastic.

References

1. Commission Decision 2005/618/EC. Off J. Eur. Comm. L., 2005, 214, 65.
2. T. P. J. Linsinger, S. Voorspoels, A. Liebich.; Anal. Bioanal. Chem., 2008, 390, 399.
3. R. Zeleny et al. Anal. Bioanal. Chem., 2010, 369, 1501.

	PBDE 47	PBDE183	PBB209	PBDE206 ^a	PBDE 209
Certified value (g/kg)	0.245	0.087	0.748	0.030	0.78
Certified uncertainty (g/kg)	0.023	0.008	0.08	0.011	0.09
Mean obtained values (g/kg)	0.259	0.081	0.725	0.021	0.793
Standard deviation, n = 6 (g/kg)	0.006	0.006	0.034	0.001	0.021
Mean recovery (%)	105.7	93.1	96.9	70.0	101.6
Standard deviation (%)	2.4	6.8	4.5	3.3	2.7

Table 2. Results for BFRs in ERM-EC591. ^aNot a certified value in EC591 [3] and not a certified uncertainty but s (n = 4).

Agilent ICP-MS User Wins Poster Prize at Nordic Plasma Conference

Raimund Wahlen
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Our congratulations go to PhD student Veronika Sele (pictured) from the National Institute of Nutrition and Seafood Research (NIFES) in Bergen, Norway. Veronika won 1st prize at this year's biennial Nordic Conference on Plasma Spectrochemistry, held in Loen, Norway, for her poster on the determination of arsenolipids in fish oils. Veronika carried out the study at the Technical University of Denmark (DTU), Copenhagen, where she used an Agilent 1200 series HPLC system coupled to a 7500ce ICP-MS.

Stronger Together: Varian, Inc. is Now Part of Agilent Technologies

Agilent is known for developing state-of-the-art technology and exceptional products. With the 2010 addition of Varian Inc., we now offer an unmatched range of instrumentation as well as the most comprehensive columns and supplies portfolio in the market. Just as important are the best-in-class service and technical support teams with deep expertise focused on finding solutions for our customers.

Our customers are engaged in a broad range of endeavors, from performing routine tests to addressing complex scientific challenges that will impact the way we live. At Agilent, we understand that all scientists, regardless of their specific area of focus share a common need for answers they can trust. This universal need is summed up in a word: **Confidence**.

Confidence means having tools that are precise, consistent, and utterly reliable. It means access to expert support to ensure that those tools run with optimal performance. Above all this means knowing Agilent, now with the strength of the people and products from Varian, provides the technology – and the measure of confidence – you need to be successful. Learn more at:
www.agilent.com/go/varian

Take a look at page 2 and 3 for details of Agilent's range of atomic spectrometry products.

Deadline Approaching for Agilent Speciation Handbook Submissions

Half page abstracts are being accepted for the 2nd edition of the Speciation Handbook **ONLY UNTIL 30 Sept, 2010**. Please submit your abstract via the web site at: www.agilent.com/chem/speciationhandbook from Sept 1, 2010. **Apple iPads** will be awarded to 5 submissions according to published contest rules. Contact Steve Wilbur with any questions at steven.wilbur@agilent.com.

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New 7700 Series ICP-MS Maintenance Videos Available on DVD

Tomo Yamada ICP-MS Product Manager, Agilent Technologies



The 7700 Maintenance Video DVD contains over 2 hours of video divided into 43 short clips that help you perform common maintenance procedures for the Agilent 7700 Series ICP-MS. The videos explain each step in detail, including information about necessary tools and parts. The DVD is available in English and Japanese, and the English script is also included for reference. A copy of the DVD is now shipped with every instrument. It will also be available to order from your local Agilent contact from September 1, 2010, at minimum cost, P/N: G3280-60910.

Conferences. Meetings. Seminars.

JAIMA Expo 2010
Sept 1-3, 2010,
Makuhari Messe, Japan
www.jaimasis.jp/2010/english

4th Asia-Pacific Winter Conference on Spectrochemical Analysis
Nov 26-30, 2010, Chengdu, China

37th FACSS
Oct 17-21, 2010,
Raleigh, NC, USA
www.facss.org

European Winter Conference on Plasma Spectrochemistry
Jan 30-Feb 4, 2011, Zaragoza, Spain
www.winterplasmazaragoza2011.es

Agilent ICP-MS Publications

To view and download the latest ICP-MS literature, go to www.agilent.com/chem/icpms and look under “Library Information”

- **Brochure:** 7700 Series ICP-MS. Extraordinary design. Unparalleled performance. 5990-4025EN
- **Application Note:** Using Qualifier Ions to Improve ICP-MS Data Quality for Waste Water Analysis, 5990-5890EN
- **Application Note:** Basic Performance of the Agilent 7700s ICP-MS for the Analysis of Semiconductor Samples, 5990-6195EN
- **Advertorial:** Collision/Reaction Cells in ICP-MS: Cell Design Considerations for Optimum Performance in Helium Mode with KED, 5990-5955EN
- **Advertorial:** Agilent's Atomic Spectrometry Family, 5990-6287EN

The following **Scientific Posters** are also freely available online. Go to the **Poster** section of the **ICP-MS Literature Library** or use the **Search** facility to search by title.

- ICP-MS: The Ultimate GC Detector

Agilent ICP-MS Journal Editor

Karen Morton for Agilent Technologies
e-mail: icpms@agilent.com



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