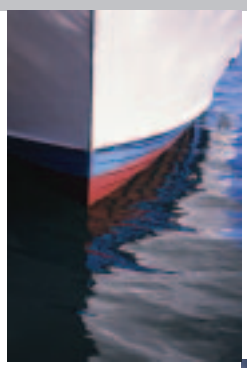
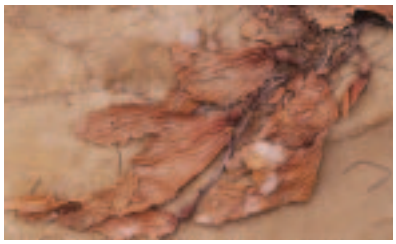
Elemental speciation analysis is now readily achieved with Thermo Electron Corporation by coupling gas chromatography to the X Series ICP-MS. The unique X Series dual mode sample introduction system and PlasmaLab software facilitate automated GC-ICP-MS performance testing, tuning and rapid switching between total element and speciation analyses.

X Series ICP-MS:

Coupling the X Series Quadrupole ICP-MS with Gas Chromatography for Elemental Speciation Analysis





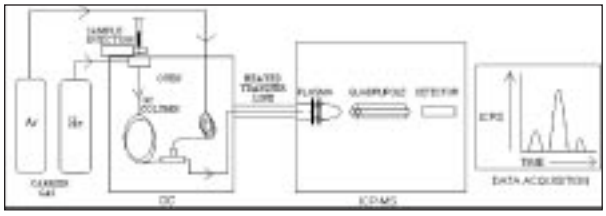


Figure 1: Block diagram of GC-ICP-MS set up

ICP-MS is a rapid and highly sensitive multi-element analytical technique, used routinely for quantification of total analyte concentrations in environmental, clinical, geological, semi-conductor, nuclear and speciality chemical matrices. However, the measurement of total element concentrations provides no information about the chemical species of the elements in the sample. To date there is increasing interest in elemental speciation analysis because each elemental species exhibits unique physical properties such as solubility, boiling point, toxicity and metabolic pathways in living organisms.

An effective analytical approach to enable separation and quantification of volatile elemental species is to couple Gas Chromatography (GC) with ICP-MS instrumentation. This coupling is achieved by connecting the outlet from the GC column to the torch of the ICP-MS using a temperature controlled transfer line as shown in Figure 1.

GC and ICP-MS techniques can be coupled effectively to enable highly sensitive and selective separations of elemental species in liquid or gaseous samples. The GC instrumentation facilitates sample introduction and volatilisation of the solvent matrix prior to separation of the required elemental species in a flow of gaseous temperature controlled mobile phase. Elemental species are separated in accordance with boiling point characteristics and their affinity for the stationary phase component. Separated species are then swept through the temperature controlled transfer line by the gaseous mobile phase and are introduced to ICP-MS. Separated elemental species are processed in the plasma and concentrations

of the elemental species constituents are quantified through the acquisition of transient time resolved signals. The advantages of using this coupled technique are being increasingly recognised for elemental speciation analysis in environmental and biological samples to assess direct toxicological risks to biota. Some typical examples of GC-ICP-MS applications include the analysis of butylated/phenylated Sn and methylated/ethylated Hg species in contaminated waters, leachates and soil/sediment extracts.

GC-ICP-MS Coupling Packs for X Series ICP-MS:

GC-ICP-MS coupling is easily achieved when using the X Series ICP-MS because all electrical and analytical connections are established using the new GC-ICP-MS Coupling Pack (P/N 4600503). The X Series ICP-MS is configured with a dual mode sample introduction system to enable simultaneous introduction and analysis of liquid and gaseous samples. The GC is then connected to the X Series using the temperature controlled GC Transfer Line and Power Supply Unit. The key components of this unique sample introduction system include the facility for simultaneous connection of both the temperature controlled GC transfer line and the nebulizer/impact bead spray chamber configuration (Figure 2) using the three legged X Series ICP-MS torch shown in Figure 3.

The dual mode sample introduction system allows analysis of aqueous multi-element solutions for X Series tuning and optimization whilst operating in the GC-ICP-MS configuration. This unique hardware feature enables efficient characterization of GC-ICP-MS instrument performance using the PlasmaLab Performance Test facility and automated GC-ICP-MS tuning and optimization is also achieved readily, when required, using the PlasmaLab Autotune facility. The unique dual mode sample introduction system provides robust plasma conditions for analysis of organic samples by GC-ICP-MS without the necessity for addition of oxygen to the plasma and use of Pt tipped interface cones. Furthermore, rapid switching is enabled between total element and speciation analyses without making any changes to the sample introduction system.

Automated bi-directional accessory control for enhanced productivity:

PlasmaLab software for X Series ICP-MS will control any third party accessories which can give and receive contact closure or specific voltage trigger signals to allow a fully automated and productivity enhancing 'rack and run' analysis approach. Examples of such accessories include the Finnigan™ FOCUS™ and Finnigan™ TRACE™ GC instrumentation from Thermo Electron. Third party accessory control is readily achieved by configuring the external trigger card in the X Series ICP-MS PC to enable bi-directional communications between PlasmaLab and the required accessories. The external trigger card is supplied complete with all electrical connections in the GC-ICP-MS Coupling Pack. The resultant bi-directional communications provide failsafe operation for the GC-ICP-MS instrumentation. For example, the GC autosampler can be triggered to abort the analytical run in the event of a hardware or software error to prevent further unwanted sample injections and the subsequent loss of precious samples.

Third party accessory functions are harmonised effectively with X Series ICP-MS data acquisition using PlasmaLab Accessory Control Language (ACL) scripts. These scripts provide a flexible and readily adaptable approach to co-ordinate X Series ICP-MS acquisition commands and external trigger commands. A typical PlasmaLab ACL script for GC accessory control is presented in Figure 4.

Figure 2: Dual sample introduction system

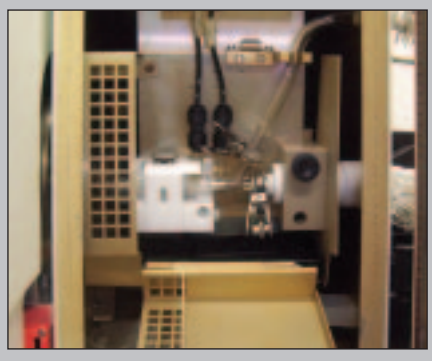


Figure 3: Three legged X Series ICP-MS torch

Flexible data acquisition and processing for Transient TRA:

Transient TRA (Time Resolved Acquisition) data can be acquired following analyte selection and definition of the required Acquisition Parameters within the Transient TRA experiment and analytical templates can be customised and stored within PlasmaLab for routine speciation analysis. The Acquisition Parameters tab allows individual analyte dwell times to be defined and the Sample List tab provides a simple format for defining analytical run times and sequencing automated sample analysis. Two modes of internal standardisation may also be used simultaneously when acquiring Transient TRA data within the X Series PlasmaLab software to improve the analytical data quality. The dual mode sample introduction system facilitates continuous analysis of aqueous internal standards throughout the GC-ICP-MS analytical run. This unique hardware facility can be used in conjunction with PlasmaLab Timeslice TRA internal standard facility to correct for baseline drift during sample analysis with a temperature ramped GC oven program and the Transient TRA internal standard facility enables transient peaks to be quantified as internal standards to correct for losses during sample preparation. An example of the simultaneous Timeslice TRA and Transient TRA internal standards facility is shown by the chromatograph in Figure 5.

PlasmaLab Transient TRA data is automatically stored in the Numerical Results tab of the PlasmaLab software and the associated chromatographic data is plotted automatically in the Acquisition Parameters tab. An intelligent peak searching facility allows integration of the raw data within the PlasmaLab software and chromatographic peaks can be integrated and identified in accordance with user defined Peak Search windows. These search windows are superimposed over the raw analytical data to allow the user to check and re-integrate the analytical data as required as shown in Figure 6.

The Numerical Results tab automatically displays integrated counts per second (ICPS) for transient peaks following assignment of the peak integration parameters and unknown analyte concentrations can be quantified in PlasmaLab using fully quantitative calibration methods. Fully quantitative concentration data is displayed in the Numerical Results tab and some example calibration data is shown in Figure 7.

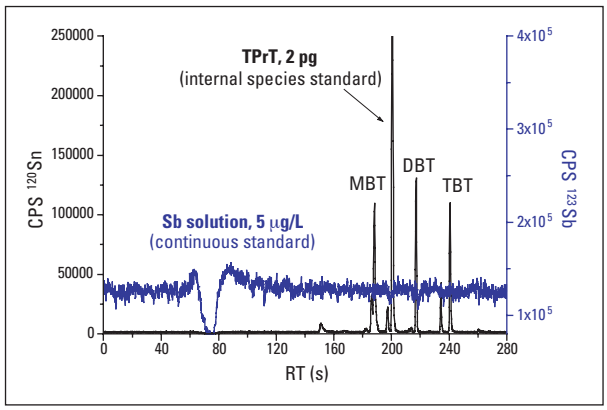


Figure 5: Chromatograph showing simultaneous Timeslice TRA and Transient TRA internal standard facility (TPrT: tri-propyl Sn species)

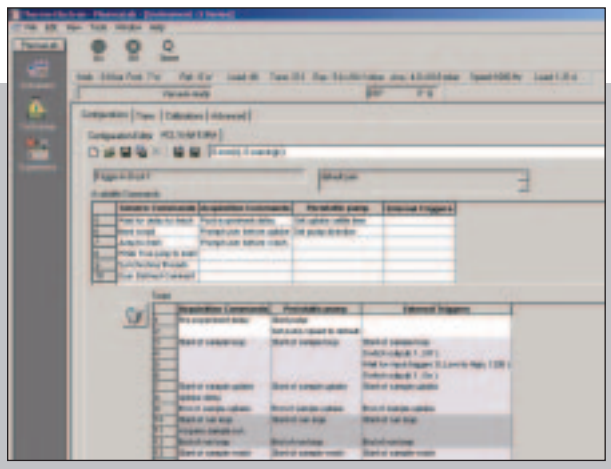


Figure 4: PlasmaLab ACL script for GC accessory control

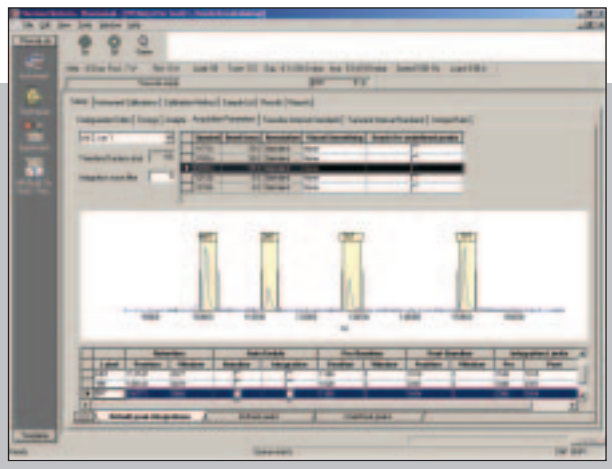


Figure 6: PlasmaLab acquisition parameters showing chromatography for mono-, di-, tri-butyl Sn and tri-phenyl Sn species (MBT, DBT, TBT and TPT, respectively)

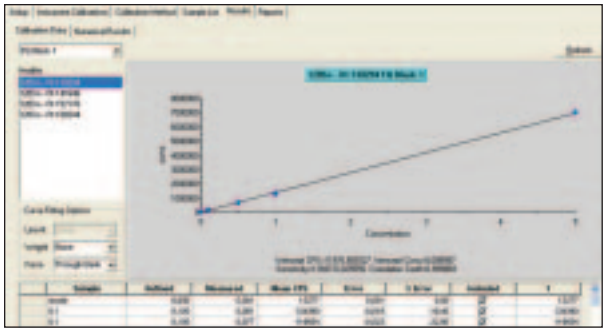


Figure 7: Fully quantitative calibration for Sn species using integrated PlasmaLab Transient TRA data

Reports:

The X Series PlasmaLab software provides a flexible approach towards data reporting and automatically presents transient TRA concentrations, integrated counts per peak and raw timeslice TRA data. Analytical results are displayed and printed in XML format to allow integration with LIMS systems and third party software packages and data can also be exported easily in CSV format if required.

Conclusions:

Elemental speciation analysis is readily achieved by coupling gas chromatography (e.g. Finnigan FOCUS or Finnigan TRACE GC) to the X Series ICP-MS. The unique dual mode sample introduction system of the X Series enables automated performance testing and optimization for the GC-ICP-MS system and rapid switching between total element and speciation analyses without re-configuring the sample introduction system. The X Series PlasmaLab software enables automated control of any GC accessory that supports hardware triggering using bi-directional communications, and both Timeslice and Transient TRA internal standard modes can be used simultaneously to improve data quality. PlasmaLab offers flexible chromatographic peak integration facilities to enhance productivity within the standard software package, providing a routine analytical solution for elemental speciation.

Features:

- Unique dual mode sample introduction for simultaneous analysis of gaseous and liquid samples and rapid switching between total element and speciation analyses.
- Flexible control of GC accessories using bi-directional communications.
- Integrated software facilities for chromatographic peak integration.
- Two internal standardisation modes to enable automated baseline drift correction (Timeslice TRA) and monitoring of instrumental drift (Transient TRA).
- Flexible reporting for raw Timeslice and integrated peak data.

Parts List:	Part Number
GC-ICP-MS Coupling Pack	4600503
-Advantech PCI Trigger Card	
-7/02 Electrical Wire (2.0 m)	
-37 Pin D-Type Male Connector	
-X Series dual sample introduction system	
-GC Transfer Line	
-Power Supply/Temperature Control Unit	
-Transfer Capillary MXT Guard Column (0.28 mm id)	
-3/16" wrench tool for MXT connection	
-Ferrules/Reducing Unions for analytical connections	
-Coiled SS Capillary, 1/16" od, 1.0 mm id	

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Thermo Electron Limited, Winsford, UK is ISO certified

Australia +61 2 9898 1244
Austria +43 1 333 50340
Belgium +32 2 482 30 30
Canada +1 800 532 4752
China +86 10 5850 3588
France +33 1 60 92 48 00
Germany +49 6103 4080
Italy +39 02 950 591
Japan +81 45 453 9100
Netherlands +31 76 587 98 88
Nordic +46 8 556 468 00
South Africa +27 11 570 1840
Spain +34 91 657 4930
Switzerland +41 61 48784 00
UK +44 1442 233555
USA +1 800 532 4752

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