A commercial perspective on the growth and development of the quadrupole ICP-MS market†

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Inductively coupled plasma mass spectrometry (ICP-MS) has grown rapidly since its commercial introduction in 1983 to become the premier technique for trace metals analysis. While the researchers in academia that pioneered the technique of ICP-MS have become well known, virtually nothing has been written about the role played by scientists working in the instrument companies that commercialized the technique. Certainly the speed of development in the early years of ICP-MS was phenomenal, when viewed retrospectively. In 2006, ICP-MS as a technique has matured: all currently available mainstream ICP-MS instruments perform well for a range of applications and there are few obvious areas where new developments are urgently required. Yet if we consider that the first ICP-MS paper was published in 1980 (R. S. Houk, V. A. Fassel, G. D. Flesch, H. J. Svec, A. L. Gray and C. E. Taylor, *Anal. Chem.*, 1980, **52**, 2283–2289), it is incredible to conceive that the first commercial ICP-MS was introduced just 3 years later at Pittcon in 1983, and that the first benchtop ICP-MS was introduced only 11 years after that. This is an account of the work of the manufacturer R&D teams responsible for the development of commercial ICP-MS in the early years, and the design features of the early instruments.

Introduction

This article begins with a review of the commercial origins of the technique and in particular the events surrounding the commercial introduction of the first two ICP-MS instruments. Developments leading to second generation instruments and the advent of benchtop ICP-MS is also discussed. A detailed model history of all ICP-MS manufacturers is presented, and finally some comments on the future of ICP-MS are included.

Origins of Sciex and VG ICP-MS

The first two companies to commercialize ICP-MS were Sciex in Canada and VG in the UK. Sciex was an MS company formed in the 1970s in Toronto by Neil Reid, Barry French and Adele Buckley. In 1981, an existing project at Sciex, the coupling of a microwave induced plasma (MIP) to a quadrupole MS was changed into an ICP-MS project under Don Douglas, following the publication of the first paper on ICP-MS by Sam Houk *et al.* at Iowa State University in 1980.[1] By the end of that year a prototype had been built (Fig. 1). As can be seen from the photograph, even at that very early stage, the prototype looked well advanced: the rail mounted torch box can be seen on the right hand side, interfaced to the cylindrical cryoshell and vacuum chamber on the left. The first data presented at the Plasma Winter Conference in January 1982 and the world's first commercial ICP-MS—the Sciex ELAN 250—was introduced at Pittcon in March 1983.[2] A co-author of Houk's seminal 1980 paper on ICP-MS was Alan Gray of the University of Surrey in the UK. Gray was the first person to interface a DC plasma with MS in 1974 while he was working for ARL in the UK. Gray later spent time working with Velmer Fassel and Sam Houk at Iowa State University (Houk was a graduate student of Fassel's) in late 1978 and began his own project to build an ICP-MS instrument around that time. The University of Surrey instrument is shown in Fig. 2. This first version employed a conventional ICP-OES vertical plasma: the sampling cone can be seen at the top of the torch box, angled down towards the plasma. The torch box was later rotated through 45 degrees and then finally 90 degrees in search of increased sensitivity. At that time, VG Isotopes, part of the UK-based VG Instruments group and led by Barry McKinnon, manufactured thermal ionization MS and glow discharge MS, and took an

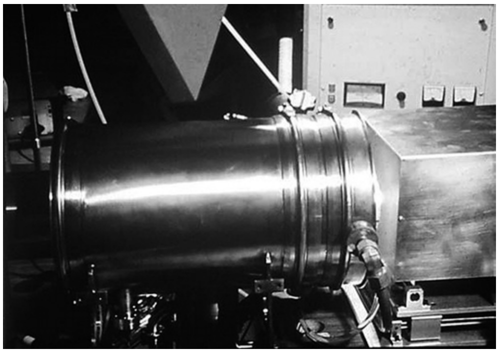

Fig. 1 Sciex ICP-MS prototype 1981. Photograph provided by Scott Tanner.

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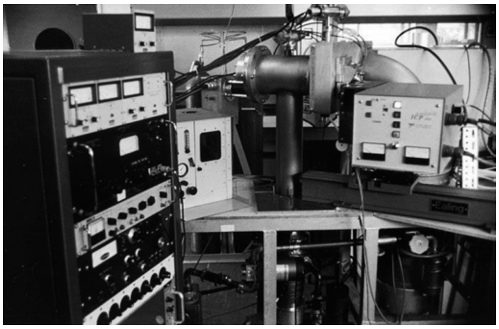

Fig. 2 ICP-MS prototype at the University of Surrey, UK, *c*1979. Photograph provided by Kym Jarvis.

interest in Gray's work. McKinnon started a project to commercialize ICP-MS, based on the work of Alan Gray and later Alan Date at the British Geological Survey. This led to the development of the VG PlasmaQuad which made its introduction at Pittcon in 1984. The world's first commercial ICP-MS order was placed shortly after and in September 1984 the first PlasmaQuad was delivered to the UK Ministry of Agriculture, Fisheries and Food (MAFF).

The early years: 1983–1993

Sciex and VG shared the ICP-MS market for several years after the introduction of the Sciex ELAN 250 in 1983 and the VG PlasmaQuad in 1984. Compared to the established metals techniques of atomic absorption spectroscopy (AAS) and ICP emission spectroscopy (ICP-OES), ICP-MS instruments were big, expensive, difficult to use and unreliable, but they had a lot of potential: the established techniques could not match the specificity or sensitivity of ICP-MS—especially for the rare earth elements and the actinides. Geological, nuclear and general research labs were quick to invest in ICP-MS, and from that initial user base the ICP-MS market grew, despite the limitations and poor reliability of the first generation instruments. The market was still served, however, by two relatively small companies, each with a strong focus on research users and with little experience of building instruments for routine labs, or providing the level of support that routine users demanded. The marketing agreement between Sciex and PerkinElmer (PE) in 1987, in which PE took over responsibility for sales, marketing and support for Sciex ICP-MS worldwide, changed the competitive landscape. PE brought its extensive experience of building and supporting instrumentation for the routine lab and as a leading manufacturer of AAS and ICP-OES was well known in the metals analysis market.

VG rose to the challenge and formed VG Elemental, a dedicated ICP-MS company, and introduced the PQ2 in 1988 which had many major improvements (much smaller cabinet, better RF generator, dual mode detector) over the original PlasmaQuad. The first PE/Sciex branded instrument was the ELAN 500, but the ELAN 5000 (1989) was the first completely new instrument produced after the PE/Sciex agreement. The 5000 was aimed at the routine market (smaller, easy to use, minimal ion lens tuning, turbopumps) and competition between the 5000 and PQ2 was intense between 1989 and 1993. By 1993, ICP-MS was firmly established as a metals technique and had begun to be adopted relatively widely by routine labs. The leap in performance, usability and reliability between the original commercial instruments and the second generation ELAN 5000 and VG PQ2 instruments cannot be overstated, and the innovation and ingenuity of the respective R&D teams has never been recognized.

It was indeed a very exciting time to be working for an ICP-MS instrument manufacturer. Where PE/Sciex focused on robustness and usability, aiming at the routine environmental and clinical market (both markets PE knew well), VG Elemental's focus was on highest performance and innovation. VG's customer base was research, geochemistry and the nuclear industry. In 1986, Chris Tye at VG developed the first commercial laser ablation system for ICP-MS (VG LaserLab) which enabled VG to successfully develop the laser ICP-MS market worldwide. In 1988, VG also introduced a nuclearized version of the PlasmaQuad which, when fitted with an enclosure could be used in nuclear hot labs. R&D activity at VG Elemental proceeded at a furious rate: following the 1988 introduction of the PQ2, in 1989 VG introduced the PlasmaTrace —the world's first magnetic sector high resolution ICP-MS, and in 1990 the PQe—an ICP-MS aimed at the ICP-OES market. The PQe had some radical design features including PC control, very small size (though still floor standing), and a Faraday detector. The detector didn't require vacuum to be maintained in the system when not in use, so a vacuum isolation valve was not necessary, and a two-stage vacuum system using a single turbopump could be used, saving space and cost. The PQe was ahead of its time, since at that point ICP-MS was not fully accepted across the environmental industry. Also, its detection limits were only marginally better than the best offered by ICP-OES instruments, so ICP-OES users didn't have sufficient reason to switch from ICP-OES to the PQe, and the PQe failed to gain wide acceptance.

The entry of HP: 1994 onwards

In the early 1980s, Yokogawa Electric, a Japanese instrumentation company, distributed Instrumentation Labs' ICP-OES in Japan. Yokogawa staff were impressed by the ELAN 250 at Pittcon in 1983 and decided to start a project to build their own instrument. With very little technical information, and no relationship with academic groups involved in ICP-MS, the Yokogawa R&D team built their first prototype in September 1983. That instrument suffered from severe interface discharge and never worked successfully. Yokogawa staff visited Pittcon again in 1984, saw the PlasmaQuad, and took back a PlasmaQuad brochure to Tokyo. On the back page of that brochure was a picture of the ICP-MS prototype at the British Geological Survey; from that photograph, Yokogawa R&D engineer Ken Sakata built a second prototype (Fig. 3) which produced the first ICP-MS data in Japan in September 1984. Three years later, in 1987, Yokogawa introduced the PMS100, which had some novel features that did not feature on other instruments for some years: it was small, controlled completely from the PC (no knobs or buttons on the mainframe cabinet) and used an off-axis ion lens design, giving vastly superior signal to noise compared to the on-axis/photon stop designs used by all previous instruments. Only one PMS100 was produced and it shipped to NIES in Tsukuba.

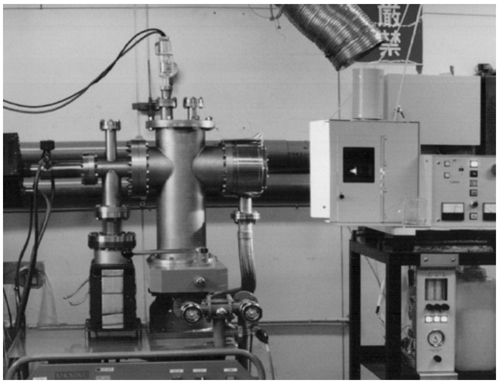

Fig. 3 Yokogawa ICP-MS prototype 1984. Photograph provided by Ken Sakata.

It was decided that some aspects of the performance of the PMS 100 were not good enough and an all-new PMS200 was introduced in 1988. Yokogawa achieved good success in Japan with further innovations such as cool plasma,[3] and in 1992 a new joint venture company Yokogawa Analytical (YAN) was formed with Hewlett–Packard (who had a long relationship with Yokogawa), to produce an ICP-MS for the world market. In two years, YAN designed and built the HP4500, the world's first benchtop ICP-MS, which was introduced at Pittcon in 1994. The HP4500 was significantly smaller than any previous ICP-MS and had features such as full PC control, automated torch positioning, off-axis ion lens and a 3 MHz quadrupole which were new to the market outside Japan, but perhaps the most important feature was its cool plasma analysis mode. Cool plasma opened up the semiconductor market to ICP-MS, and many semiconductor labs switched from GFAAS (graphite furnace atomic absorption spectroscopy) to cool plasma ICP-MS with the HP4500.

ICP-MS model history and market

Fig. 4 shows the timeline of every ICP-MS model introduced from 1983 to 2006. During that time there have been many company name changes, mergers and consolidations, but what is fascinating is the number and variety of models that have been produced. ICP-MS is certainly one of the most dynamic markets in the analytical instrumentation business. Quadrupole ICP-MS (ICP-QMS) remains by far the largest instrument type, with approximately 95% of all ICP-MS sold being ICP-QMS and the remainder being magnetic sector type. Time-of-flight (TOF) MS analyzers have been applied to ICP-MS but have not been widely accepted, and for a period Hitachi marketed a MIP-MS in Japan.

The future of ICP-MS

When ICP-MS was introduced in 1983, ICP-OES was itself only 9 years old and had just become established as a mainstream metals technique alongside AAS. ICP-OES continued to grow strongly over the next few years but the market has now levelled out and, of the three metals techniques, only ICP-MS is showing significant growth, while the AAS market is in decline. To 2006, twenty three years have passed since the introduction of ICP-MS but there is no new trace metals analysis technique on the horizon to challenge it. ICP-MS as a technique has matured and, except for the notable exception of collision/reaction cells, there have been few significant advances in the technology in

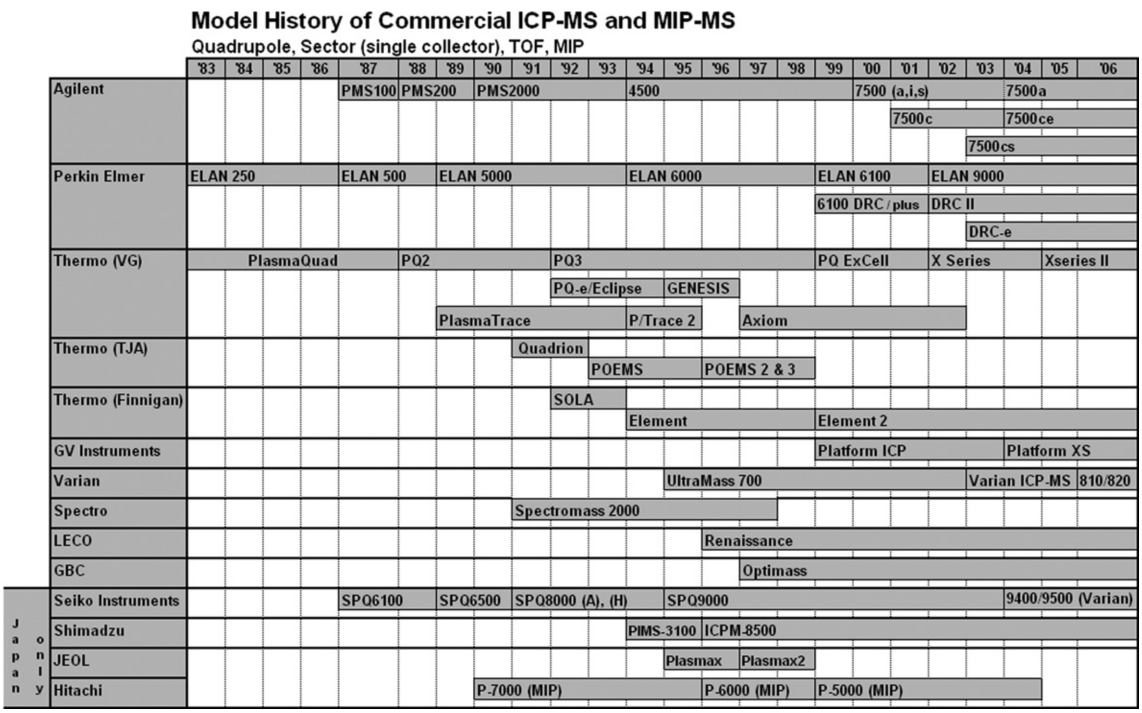

Fig. 4 Timeline of commercial ICP-MS instruments.

recent years. ICP-MS will not completely replace ICP-OES in the foreseeable future but the outlook for ICP-MS remains very positive for many years to come.

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