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TRENDS

Inductively coupled plasma mass spectrometry: recent trends and developments

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Abstract This year inductively coupled plasma mass spectrometry (ICP-MS) moves into the fourth decade of development. In this article, some recent trends and developments in ICP-MS are reviewed, with special focus on instrumental development and emerging applications. Some key trends include a novel mass spectrometer for elemental and speciation analysis in Mattauch–Herzog geometry with a focal-plane-camera array detector. The reason for this development is the possibility to record the full elemental mass range simultaneously and all the time. Monitoring fast transient signals in chromatography or laser ablation is now possible and will become an important asset in future studies, e.g., for isotope ratio analysis. In addition, there is a lot of new activity and interest in the area of nanosciences and medicine. Here, instrumental developments are reported that allow the direct analysis of microparticles and single cells.

Keywords Inductively coupled plasma mass spectrometry · Trends · Ultratrace analysis · Simultaneous detection · Array detector · Time of flight · Nanoparticles · Single-cell analysis · Laser ablation

Introduction

The first mass spectra obtained with an inductively coupled plasma (ICP) mass spectrometer were seen on 6 June 1978 [1]. Robert Samuel Houk, at that time a graduate student in the Velmer A. Fassel laboratory at Iowa Sate University, worked together with visiting scientist Alan L. Gray and laid the groundwork for the first publication on ICP mass spectrometry (MS) in 1980 [2]. Gray [3–6] had already demonstrated the benefits of plasma-source MS for elemental analysis but with a wall-stabilized direct-current (DC) plasma in argon. Thirty years later, the first ICP-MS paper has been cited 369 times and more than 16,000 manuscripts have been published on original research covering fundamentals and applications of ICP-MS (according to a search conducted with ISI Web of Knowledge on 9 September 2010; see Fig. 1).

At the beginning of the fourth decade of development, ICP-MS stands as a useful tool with a wide range of applications, not only in multielement analysis. Just recently, a number of excellent reviews were published that covered fundamental studies of ICP-MS [7] and applications in the fields of speciation analysis [8], metallomics, proteomics [9, 10], bioimaging [11], nanoscience [12], and many more [7]. Garcia et al. [13] discussed the current status of laser ablation ICP-MS and offered an outline of possible future developments. Vanhaecke et al. [14] provided a tutorial review on single-collector and multicollector ICP-MS for isotopic analysis. Hieftje [15] considered how analytical instruments and methods are being developed for tomorrow's needs. In 2010, a series of events were organized to celebrate 30 years of ICP-MS. For example, a full-day special symposium sponsored by the *Journal of Analytical Atomic Spectrometry* was held at the 37th Federation of Analytical Chemistry and Spectroscopy Societies meeting in Raleigh (NC, USA). Speakers from around the world highlighted the development and importance of past and future ICP-MS.

This article highlights trends and developments in ICP-MS, with special focus on instrumental development and

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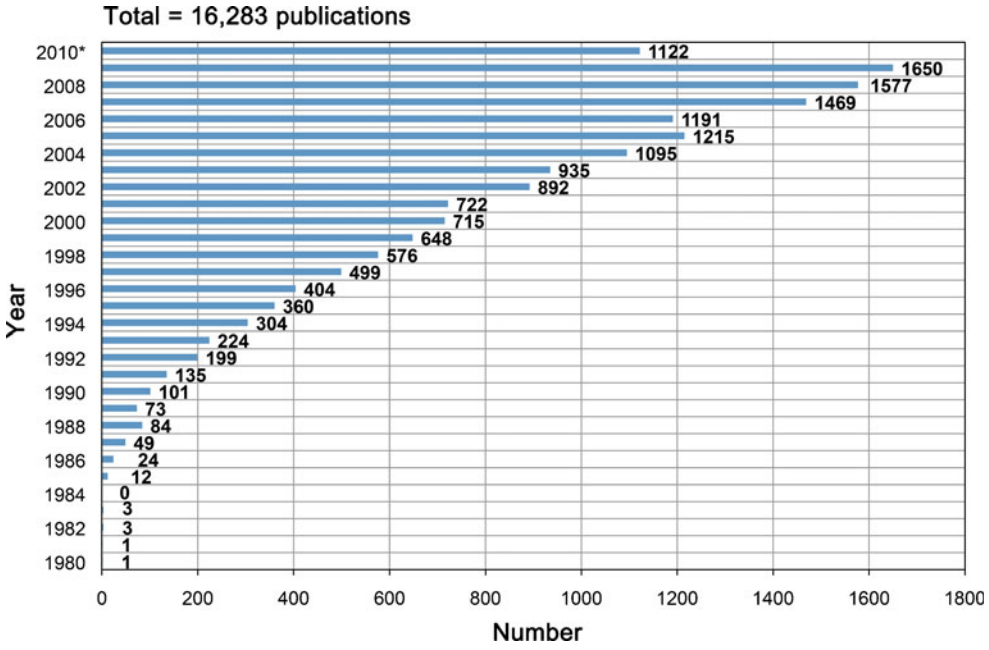

Fig. 1 Number of inductively coupled plasma mass spectrometry (ICP-MS) publications by year, according to an ISI Web of Knowledge search conducted on 9 September 2010. The *asterisk* indicates partial results for 2010. Total of 16.283 publications. Note that the first papers on ICP-MS are not found in the ISI Web of Knowledge database using the search terms "ICP-MS," "ICP MS," "Inductively coupled plasma mass spectrometry," "Inductively coupled plasma mass spectrometry," or "Inductively coupled plasma source mass spectrometry." Those papers have been added manually to the records

emerging applications. Over the last few years, a variety of papers on fundamentals and new approaches in inorganic MS have been published. Of course, only a small selection of papers can be discussed in a trends article. The author finds some developments particularly interesting and regrets any inadvertent omission of reference to relevant papers. Most of the papers that will be discussed here have just been published.

Continuous simultaneous detection

Conventional ICP-MS instruments, namely, quadrupole and magnetic sector field instruments, are operated in a scanned manner with a low duty cycle. Only one *m*/*z* value can be measured at a time. Multicollector ICP-MS instruments are able to detect isotopes of selected elements simultaneously, but only for a small number of *m*/*z* windows. Simultaneous detection of the full elemental mass range at all times is not possible with those instruments. Here, continuous simultaneous detection offers several advantages. First, an improved duty cycle can lead to better precision, shorter analysis times, and superior limits of detection, especially if a transient is faster than the scanning speed of the mass analyzer. Second, correlated noise can be reduced by ratioing techniques, leading to enhanced isotope ratio accuracy. Finally, spectral skew, that is, the error in relative signal levels for several *m/z* values across a transient signal produced by a scanning instrument, can be eliminated. Schilling et al. [16] recently reviewed the benefits of continuous simultaneous detection.

Scientists from Indiana University (G.M. Hieftje et al.), University of Arizona (M.B. Denton et al.), and Pacific Northwest National Laboratory (D.W. Koppenaal et al.) have developed an instrument for continuous simultaneous detection. A Faraday-strip array detector, termed the focal-plane camera (FPC), was placed at the focal plane of a Mattauch–Herzog mass spectrograph (MHMS) coupled to an ICP. Four generations of FPCs have been developed and characterized: 31-channel [17, 18], 128-channel [19, 20], 512-channel [21, 22], and 1,696-channel [23, 24] Faraday-strip array detectors. The improvement of mass resolution with FPC generation is shown in Fig. 2.

The latest detector design features an array of 1,696 individual 8.5-μm-wide pixels (4-μm pixel spacing). Each pixel (Faraday strip) has its own capacitive transimpedance amplifier that incorporates two levels of gain (1:1,000), and a sample-and-hold amplifier that enables simultaneous data acquisition. The detector length is 21.2 mm and allows simultaneous continuous acquisition of signals in either the low (6–9.5 amu) or the high (150–238 amu) mass range. Eighty-eight masses can be monitored simultaneously in the latter mode. Figure 3 shows a spectrum of the lanthanide series acquired with the 512-channel detector. In principle, the available mass range is unlimited and only dictated by the size of the detector (length) and the geometry of the MHMS. However, as of today, there are economic and practical reasons for limiting the size of the detector. The analytical figures of merit of the mass spectrometer for ultratrace elemental analysis were determined with the third-generation detector and ultrasonic sample introduction. The linear dynamic range spans 8 orders of magnitude (10^{-3}–10^{5} $\mu g L^{-1}$ for Ho). Limits of detection in the single to tens of nanograms per liter range were achieved for the lanthanide series. Isotope ratio accuracy and precision of the 512-channel detector did not outperform the first- and second-

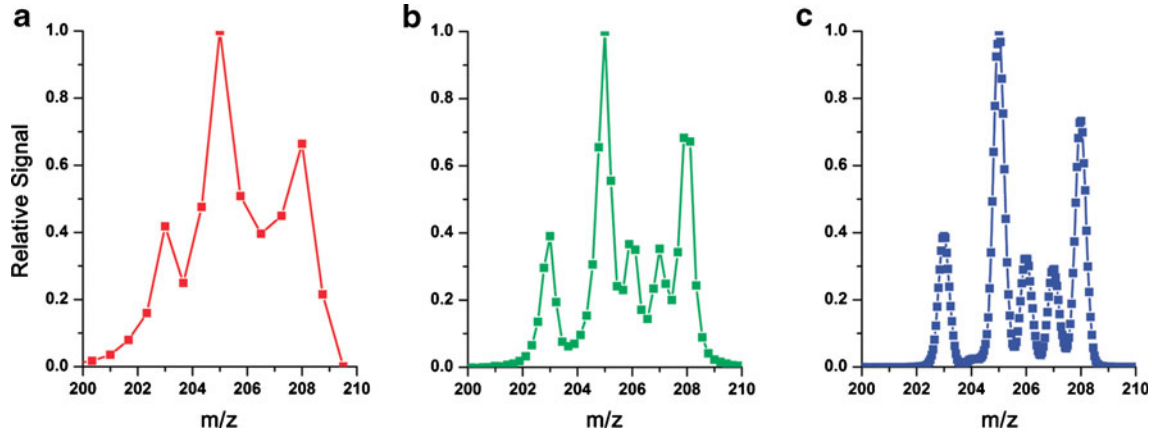

Fig. 2 Spectra of thallium and lead obtained with a simultaneously and continuously measuring ICP mass spectrometer with Mattauch–Herzog geometry and a focal-plane camera (FPC). The resolution is shown to improve with number of pixels from the 32-channel FPC (*a*), to the 128-channel FPC (*b*), to the 512-channel FPC (*c*). With the 512-channel FPC, the detector is no longer the factor limiting resolution. (Reprinted with permission from [21]. Copyright 2009 American Chemical Society)

generation detectors, most likely because of imperfect manufacturing of the semiconductor chips. However, precision levels of 300–500 ppm and accuracies of 2–10% error were obtained with 1–10-s integration times. The resolving power was 640 for ^{205}Tl for 100-μm-wide slits. More details are described elsewhere [21].

The benefits of simultaneous detection for measuring transients were demonstrated by coupling ICP-MHMS with gas chromatography [25], high-performance liquid chromatography (LC) [26], electrothermal vaporization [27], and laser ablation (LA) [28]. In the LA-ICP-MHMS, for example, correlated noise during the ablation process could be reduced by ratioing techniques, resulting in isotope ratio precision better than 0.02% relative standard deviation (RSD). Limits of detection in the tens to hundreds of nanograms per gram range were obtained with continuous ablation, whereas limits of detection in the tens to hundreds of femtogram range were found with single laser ablation shots [28]. In a later publication by the same group, isotope ratio precision values of 0.0056% RSD (ten measurements) during solution nebulization of 1 $\mu g mL^{-1}$ Ag were achieved with improved peak-integration methods [20].

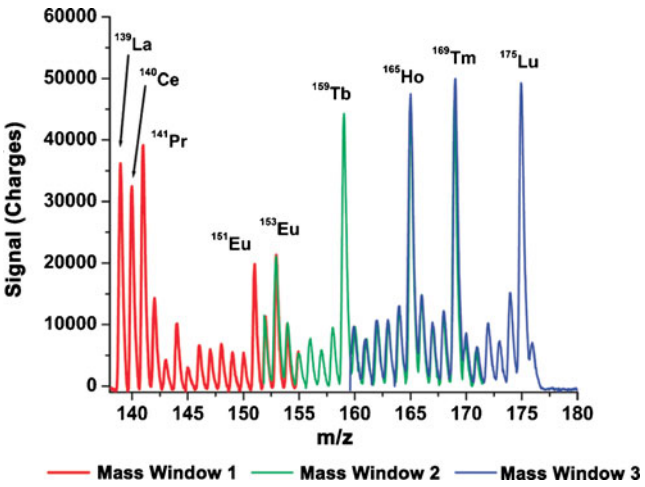

Fig. 3 Spectrum of the lanthanide series, acquired in three separate mass windows delineated by color. A 10-ppb multielement solution and 10-s integration time were used. (Reprinted with permission from [21]. Copyright 2009 American Chemical Society)

In March 2010, SPECTRO Analytical Instruments (Kleve, Germany) presented an array-detector Mattauch–Herzog mass spectrometer prototype at the 2010 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy in Orlando (FL, USA). The SPECTRO MS features an array detector with 4,800 channels and two levels of gain. Masses from 5 to 240 amu can be measured simultaneously with a 120-mm-long detector at the focal plane of the double-focusing mass spectrometer. The claimed linear dynamic range spans 7 orders of magnitude. In the first ICP-MS application report on drinking water analysis by SPECTRO [29], limits of detection in the sub-microgram per liter range (e.g., Ag, Al, As, Cd, Co, Cr, Cu, Pb, and U) to the microgram per liter range (e.g., B, Ca, Fe, K, Mg, and Na) were reported. RSDs were on the order of 0.1–0.8% for three replicates. Further, certified reference material analysis (NIST 1643e) with recoveries between 93 and 104% was reported. An initial study of the capabilities of the instrument coupled with laser ablation (GeoLasC, Coherent, Santa Clara, CA, USA) was recently presented at an ICP-MS user meeting by Grauwiler et al. [30]. At the time of this writing, however, no further data on, e.g., attainable resolution have been published.

New applications

In a 2007 trends article [31] recent ICP-MS developments were reviewed with special focus on applications in the nanosciences. Inorganic MS was successfully applied for size characterization of nanoparticles. It was also reported that ICP-MS-linked immunoassay protocols had been developed which apply gold-nanoparticle-tagged antibodies as secondary antibodies in surface-linked immunoreactions. Since 2007, the findings of a substantial number of new studies have been published that underline the potential of inorganic MS in this field. For example, researchers reported the successful analysis of individual cells with quadrupole ICP-MS [32–34], quantitation of human urinary proteins [35], immunoassay detection of α-fetoprotein in ICP single-particle mode [36], and identification of *Escherichia coli* bacteria after gold-nanoparticle-based labeling [37]. The potential use of ICP-MS with element-tagged immunoassays in direct comparison with enzyme-linked immunosorbent assays and western blotting was discussed in [38]. For more information on the use of ICP-MS in this field, the reader is directed to a review article by Wang et al. [39]. In that article, strategies and challenges for ICP-MS-based immunoassays, labeling with elemental tags, tissue imaging, as well as protein quantification are comprehensively reviewed.

As discussed in the previous section, ICP-MS technology is still limited when it comes to the detection of multiple masses in fast transients or single events and at low concentration. For example, the duration of the ion cloud in the ICP produced from a single-cell or microparticle event was found to be on the order of 100 μs [40]. Therefore, full spectra of a mass range of interest should be recorded at a frequency of 100 kHz to give at least ten points per peak. In contrast, the stabilization time of quadrupole mass analyzers is typically 50–200 μs [32]. Owing to this instrumental limitation, sequentially measuring systems are unattractive for detecting multiple ions in fast transients. Simultaneous multielement ion extraction and detection can be achieved with time-of-flight (TOF) mass analyzers [41]. Today, only one ICP-TOF-MS system is commercially available (OptiMass 9500, GBC Scientific Equipment, Braeside, Australia), after Leco discontinued its product. Single-scan full-mass spectra (m/z 1–260) can be generated in less than 34 μs with a maximum frequency of up to 30,000 spectra per second. However, only integrated mass spectra are displayed and recorded at a maximum of 50 mass spectra per second [42]. Again, single-cell analysis requires much higher frequencies of 50,000 or 100,000 spectra per second. Consequently, Bandura et al. [43] developed a fast ICP-TOF-MS instrument designed for single-cell analysis based on elemental immunoassays.

Figure 4 shows a schematic diagram of the instrument (CyTOF mass spectrometer), which is available from DVSSciences (Richmond Hill, ON, Canada). The ICP-TOF-MS instrument features a three-aperture plasma–vacuum interface [sampler (1.2-mm orifice diameter), skimmer (1-mm orifice diameter), and reducer (1.2 mm diameter)], DC quadrupole turning optics (filter for neutrals), a radio-frequency quadrupole ion guide (discrimination of dominant plasma ions), a point-to-parallel focusing DC quadrupole doublet, an orthogonal acceleration reflectron analyzer (76.8-kHz spectrum generation frequency), a discrete dynode fast ion detector, and an 8-bit 1-GHz digitizer. More details are described in [43]. The instrument can be operated in two modes, high-resolution mode and high-sensitivity mode. In high-resolution mode, mass resolution (full width at half maximum) was $M/\Delta M>900$ at m/z 159. In high-sensitivity mode, mass resolution was $M/\Delta M>500$ at m/z 159. The sensitivity (counts per second per parts per billion, less than 3% oxide ratio) for, e.g., ^{139}La was reported to be 60,000 and 100,000 in high-resolution and high-sensitivity mode, respectively. The instrument is useful, e.g., for the determination of the lanthanide series, with an available mass range of m/z 125–215 and a linear dynamic range of 5 orders of magnitude (at 1-s integration time).

The novel instrument was used for immunophenotyping, the discrimination of heterogeneous cell populations. Flow cytometry is most commonly used for immunophenotyping, but has the disadvantage of fluorescent tag emission spectral overlap, resulting in limited multiplexing capabilities. The so-called mass cytometer was successfully applied for simultaneous determination of multiple antigens and DNA in single cells. In particular, human leukemia cell lines and acute myeloid leukemia (AML) patient samples were studied. Antibodies against myeloid (CD13, CD14, CD15, and CD33), lymphoid (CD3, CD4, CD5, CD19, CD20, CD28, and CD57), nonlineage (CD34, CD38, CD117, and HLA-DR), and differentiation (CD36, CD56, CD64, CD45, and CD45RA) markers were tagged with 20 different stable lanthanide isotopes (^{139}La, ^{141}Pr, ^{142}Nd, ^{144}Nd, ^{145}Nd, ^{146}Nd, ^{147}Sm, ^{152}Sm, ^{151}Eu, ^{153}Eu, ^{156}Gd, ^{159}Tb, ^{164}Dy, ^{165}Ho, ^{166}Er, ^{170}Er, ^{169}Tm, ^{171}Yb, ^{174}Yb, and ^{176}Yb), and a novel Ir-containing metallointercalator [44] was used to measure DNA content (Fig. 5). More details on the labeling protocol are given elsewhere [44, 45]. After staining and fixation, cells were introduced into the ICP-TOF-MS instrument by pneumatic solution nebulization. Subsequently, cell-surface antigens were identified through the metal tag after data acquisition and processing with in-house-written software and flow cytometry software, respectively. Figure 5 shows polar diagrams of median intensity values from monoblastic (Fig. 5a) and monocytic (Fig. 5b) acute myeloid leukemia samples. The cell types

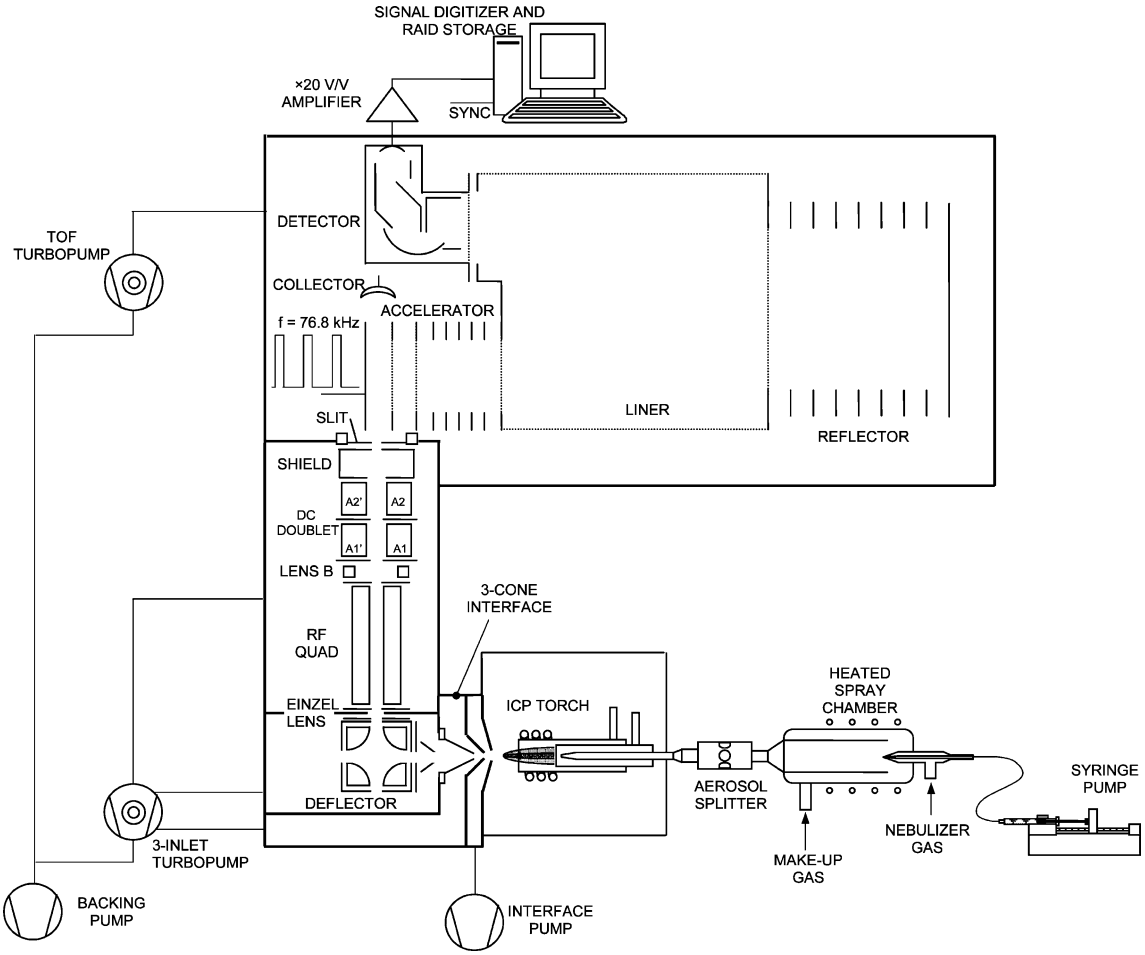

Fig. 4 The prototype CyTOF mass cytometer based on ICP time-of-flight (TOF) MS technology. (Reprinted with permission from [43]. Copyright 2009 American Chemical Society)

could be successfully discriminated and reported differences agreed well with literature values obtained with reference techniques. For more information on the instrument and applications, the reader is directed to recent publications by the group [43, 45–47]. It is anticipated that this instrument will be a helpful tool for multiplexing, cell characterization, elemental cell encoding, and element-coded bead-based gene/protein arrays.

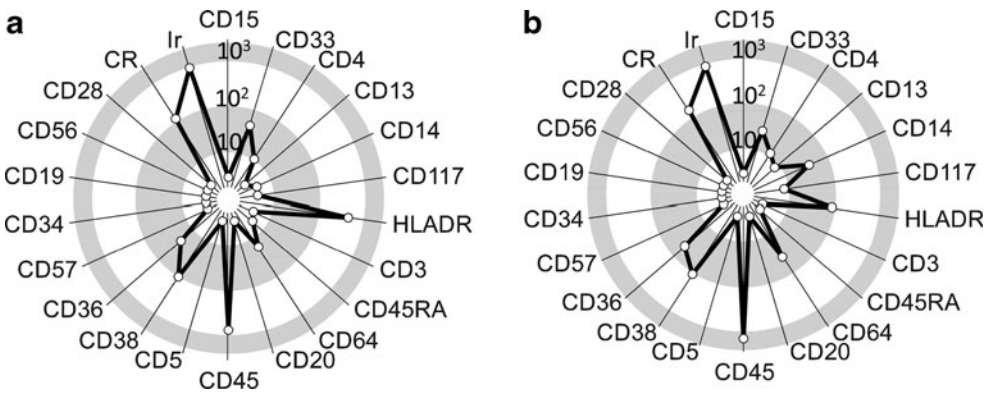

Fig. 5 Single-cell analysis with ICP-TOF-MS: polar diagrams of median intensity values for surface antigens measured using metal-tagged antibodies on leukemia patient samples: monoblastic M5 acute myeloid leukemia (AML) samples (**a**) and monocytic M5 AML samples (**b**). Each of the 22 axes represents an antibody [or contrast reagent (*CR*) or Ir–DNA intercalator] measured by detecting isotopic tags per individual cell event. The monoblastic phenotype (**a**) shows high CD33 and HLA-DR and low CD34, CD13, CD14, and CD64 expression levels. The more differentiated monocytic M5 (**b**) type displays increased CD13, CD14, and CD64 and lower HLA-DR levels. More details are given in [43]. (Reprinted with permission from [43]. Copyright 2009 American Chemical Society)

Concluding remarks and future perspectives

This article highlighted some recent trends and instrumental developments in ICP-MS. Continuous simultaneous detection of all masses between 5 and 240 amu is now possible with array-detector technology. Further improvement of the TOF technology together with labeling approaches now enables the detection of single cells or microparticles. Thirty years after its inception, ICP-MS has emerged from being a purely elemental analysis method to a versatile tool with analytical, biological, clinical, environmental, geological, and toxicological applications, and many more. ICPs are used in industry and academia around the world and approximately 600–1,000 instruments are currently sold each year. In the next 30 years of ICP-MS, however, there is still a lot to be done. In the following, a future perspective is given, meant to stimulate further development.

If we look at three major tasks in ICP-MS, plasma generation, ion sampling, and ion detection, a lot of room for improvement is apparent. For example, analytical performance heavily depends on the plasma generation with efficient coupling of the generator oscillator and the plasma load. In recent years, new type of samples with different organic matrices have been investigated since coupling of ICP-MS with LC has become a widespread tool for speciation analysis. Quantitative and species-specific analysis was successfully demonstrated with LC-ICP-MS [48]. Coupling of LC with ICP-MS is straightforward. Only a few things have to considered, e.g., use of a cooled spray chamber for the reduction of plasma load and addition of small amounts of oxygen to the nebulizer gas flow to prevent carbon buildup on the cones. However, during a gradient elution, quantitation is not possible with high accuracy owing to changing plasma conditions. Depending on the radio-frequency generator used (free-running, crystal-controlled, or solid-state), instruments show a significant, nonlinear change in total ion count with varying total organic content (TOC). This is especially true if the impedance matching network of a crystal-controlled or solid-state generator is not variable during a chromatographic run. Here, it would be required to, e.g., incorporate fast and automatic impedance matching network designs to compensate real-time variations in impedance with changing plasma conditions. Alternatively, one could investigate other means for balancing differing matrix/organic sample load in the ICP.

Ion sampling efficiency with state-of-the-art sampler/skimmer interfaces is still in the single percent range. The ICP-MS interface is conceptually unmodified from its initial design first implemented 30 years ago. Improvement of the ion sampling efficiency would presumably directly translate to improved limits of detection. Improved performance was reported by adding a second roughing pump to the vacuum system and changing sampler and skimmer cones (S-option in discontinued VG PQ ExCell instruments, jet interface option in Thermo Fisher Element 2 and Neptune Plus instruments). Limits of detection in the parts per quintillion range (5 ppqt for ^{232}Th) and improved sensitivity, e.g., approximately 200 million cps/ppb for ^{115}In, have been reported with the latter [49]. However, this approach works for dried aerosol conditions only. Clearly, there is still a lot of room for research and development. Computational fluid dynamics calculations of the Mach disk expansion between the sampler and the skimmer were recently presented by the Günther group [50]. Also, the Farnsworth group [51–53] has been involved in fundamental studies and simulations of the ICP-MS interface. Such studies will help to understand the processes taking place and hopefully lead to new interfaces and optimized ion sampling in the future.

Ion detection is typically performed with electron multipliers (discrete-dynode multipliers, channel electron multipliers) or Faraday cup detectors. Over the past 10 years or so, Hieftje and collaborators have been working on the array-detector concept. Publications from the same group demonstrate the benefits of continuous simultaneous detection. Clearly, a truly simultaneous mass spectrometer at a reasonable price will have a major impact in the field of inorganic MS. In the future, it might be useful to study the addition of a filter technology (e.g., collision/reaction cell) to the instrument for the removal of potential interferences. It is noteworthy that the array-detector concept is not limited to ICP-MS, but might find use in other areas such as ion mobility spectrometry or the recently introduced concept of distance-of-flight MS [54].

It is anticipated that key improvements to the analytical performance of ICP-MS will continue. An interesting future lies ahead for ICP-MS, with emerging applications in metallomics, proteomics, clinical diagnosis, forensics, and the nanosciences.

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