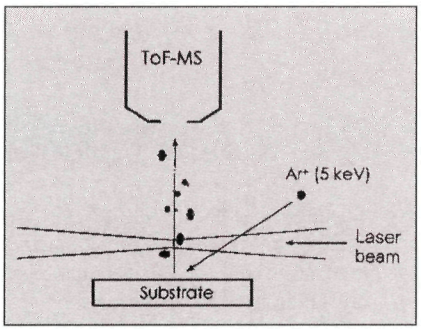


Schematic of the experimental apparatus. (Adapted with permission from MacMillan Magazines.)

Uniform elemental analysis

Although surface techniques such as secondary-ion MS can detect elements at ppb levels, they are typically semiquantitative. Chun He and colleagues from SRI International described a surface technique that provides highly uniform, quantitative, and sensitive analysis, even when the bulk material contains elements with different ionization potentials.

The method uses a steady-state 5-keV argon ion beam to sputter material from a substrate's surface and a high-intensity 2.3-eV (532-nm) laser to induce multiphoton ionization of the sputtered atoms. The photoions were then measured by TOFMS.

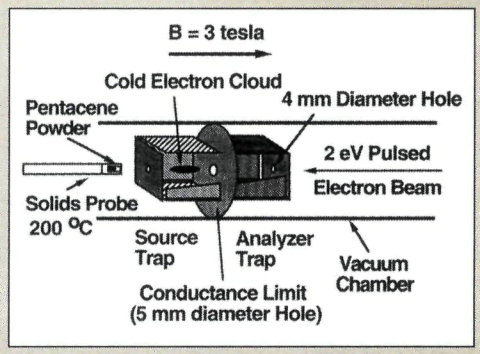
Steady-state sputtering, defined as a pulse duration of > 1 μs, avoids preferential sputtering and ensures sampling over all velocity components of the sputtered material. The photoionizing laser beam passes parallel to, and ≤ 1 mm above, the sample surface and subtends a large solid angle. Quantification is based on flux, which is equal to the number density of atoms and molecules times the velocity. The authors demonstrated the method by analyzing GaAs and SiC single crystals. (*Nature* **1997**, *385*, 797–99)

Electrons get cool

Electron-impact ionization MS is usually the method of choice for detecting and identifying environmentally important volatile compounds. However, EI-MS leads to extensive fragmentation, which reduces the molecular ion abundance and makes it difficult to distinguish between primary and fragment ions in complex mixtures. For molecules with high electron affinities, negative-ion or electron-capture MS (EC-MS) is an alternative approach that offers selective and sensitive detection—even for small amounts of compounds. One drawback is that EC-MS requires low-energy electrons, which will attach themselves to gas-phase neutral molecules.

Alan G. Marshall and colleagues at Florida State University introduced a new approach for producing these low-energy electrons by using the Penning ion trap of an FT-ICRMS instrument. Electrons emitted from a hot filament (~ 2 eV) are injected into the trap and cooled to room temperature in about 1 s by classical radiative emission. The cold-trapped electrons can then attach to neutrals with electron affinities of < 0.5 eV. Because the electrons and negative ions are simultaneously held in the Penning trap, the analytes can be detected by FT-ICRMS in the same trap.

The Florida group used a 3-T magnetic field in these experiments and formed negative ions of polycyclic aromatic hydrocarbons with electron affinities as low as 0.45 eV (pyrene). Among the advantages of this approach, say the authors, is that it avoids the use of the reagent gases needed in other EC-MS approaches, the cold electrons also cool the negative ions in the trap by sympathetic cooling, and the negative ions form only near the center of the trap and produce a compact ion packet that optimizes the FT-ICR experiment. (*J. Am. Chem. Soc.* **1997**, *119*, 2267–72)



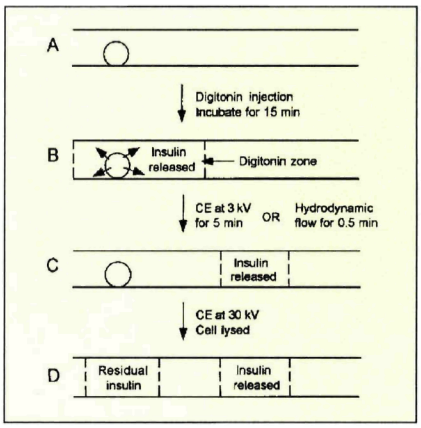
Schematic of the dual cubic Penning trap used for producing cool electrons; gas-phase neutrals are generated from a heated solids probe.

Monitoring cellular changes

Changes in cellular content reveal more about a cell's interaction with its environment than can simple quantitative measurements of intracellular components. The question is how to measure both the secreted and residual portions with a single assay. Edward S. Yeung and Wei Tong of the Ames Laboratory–USDOE at Iowa State University have developed a CE laser-induced native fluorescence method for doing just that with insulin released from single pancreatic βTC3 cells.

A single pancreatic cell was hydrodynamically injected into the end of a capillary. Digitonin, which produces pores in the cell membrane by dissolving cholesterol, was then injected into the capillary, and insulin was released. After 15 min, the insulin was separated from the cell either by electrophoresis (at 3 kV) or hydrodynamic flow. After the two zones were spatially separated, the cell was lysed by increasing the voltage to 30 kV, releasing the residual insulin. The two insulin zones could then be quantified separately with a 100-amol detection limit.

The authors also determined the amount of insulin released by digitonin in an off-column method that involved incubating and spinning down the samples. The off-column method consistently yielded higher percentages of insulin. The authors believe that the spinning process caused additional intracellular insulin to leak. (*J. Chromatogr. B* **1997**, *689*, 321–25)



Schematic of on-column monitoring of insulin release from a single cell. (Adapted with permission from Elsevier Science.)