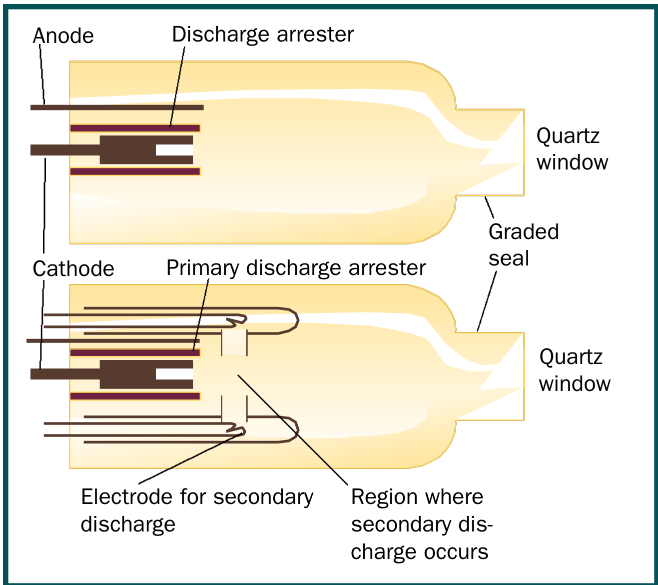
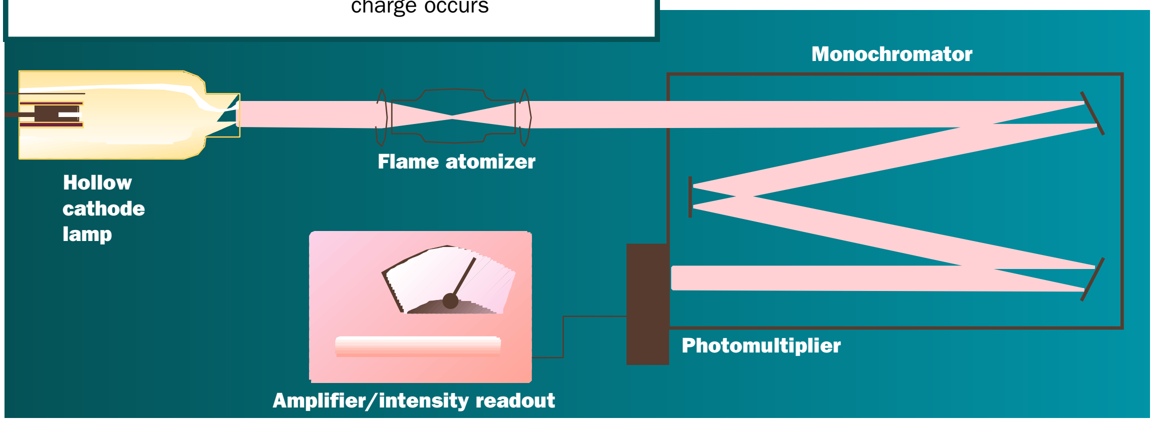
by Derek Oliver, Trevor Finlayson, and Jack Sullivan

Varian Benefits from Monash Input

Monash University and Varian Australia Pty. Ltd. (both in Victoria, Australia), are collaborating to improve hollow-cathode lamps used as emission sources in atomic absorption spectrometers. Atomic absorption spectrometry (AAS), an analytical technique for measuring the concentrations of metals and semimetals, is commonly used to check the composition of mined ores, and to monitor the levels of elements in water supplies. AAS allows elements to be measured in concentrations of parts per billion.

A casual conversation in 1992 between two of the authors, Trevor Finlayson, associate professor of physics at Monash, and Jack Sullivan, consultant to Varian, led to a preliminary project to find a method of producing a specific alloy for use in hollow-cathode lamps. The success of the project gave rise to a standing contract between Varian and the university to make rods of the alloy in the university's Department of Physics. Because Varian needs only small quantities of the high-purity alloy (a few kilograms per year)—and such small quantities are not economically viable for a commercial supplier—the contract is a good match for the specialized facilities available at Monash.





Novel mutiple-element hollow-cathode designs have been developed for both standard and boosted-output lamps (left), the emission sources in atomic absorption spectrometers (below).

Following the success of this initial collaboration, Finlayson, in association with Varian, applied for an Australian Postgraduate Award (Industry), called an APAI, to fund a graduate student to investigate other possible improvements in hollow-cathode lamps. Derek Oliver, the student who won the appointment, moved to Monash from his undergraduate institution to take up the research toward his Ph.D.

To appreciate the success of his work requires some understanding of the basic principles of AAS.

Atomic absorption

In AAS, light from a source is directed through an absorbing region, such as a flame atomizer, where a fraction of the light is absorbed by atoms in the sample. The amount of radiation that remains is then monitored on the far side of the cell by a photomultiplier tube. The most common light source is a hollow-cathode lamp, which emits the line spectrum of the element of interest. The cathode contains the element that is to be determined in the sample. For example, if copper is to be measured, then the cathode contains copper. The emission line to be used for analysis is selected with a monochromator.

The hollow-cathode lamp is filled with a low pressure of an inert gas such as argon or neon. A high-voltage discharge between the anode and the cathode of the lamp creates a plasma, or excited gas, that sputters atoms from the surface of the cathode bore. The now-gaseous atoms then undergo inelastic collisions with the plasma that raise them to excited states, re-emitting this energy as light as they relax to lower energy states. Another lamp design, called a boosted-output or high-intensity lamp, has a second discharge across the top of the cathode bore. The excitation of additional atoms in a region where there is otherwise a low probability of excitation yields a strong, almost noise-free signal characterized by a particularly sharp emission line.

The key to the design of the lamps is to produce an intense signal while avoiding line broadening, which decreases sensitivity. A higher discharge current dislodges more atoms, which in turn emit more light. The problem is that the dominant broadening mechanism is self-absorption, the absorption of the emitted light by atoms in its path, and this broadening mechanism also depends on the number of sputtered atoms. (Maximum absorption occurs at the center of the emission line, and the resulting decrease in the maximum intensity effectively increases the full width at half maximum, the conventional measure of the breadth of the line.) The balance between these competing parameters has to be struck and the performance of the lamp is optimized by adjusting the filler gas pressure, the bore geometry, and the cathode composition.

Oliver's Ph.D. work concerns the spatial distribution of sputtered atoms in the body of the lamp. For these studies, the flame atomizer is replaced by a sputtering cell, and a fine beam from an emission source is passed through the cell. Absorption readings are then made in a raster pattern as the sputtering cell is moved through the beam. Based on these characterization studies and theoretical understanding of the sputtering process and the transport of the sputtered atoms through the cathode bore, Oliver is deriving a working model of the cathode lamp that can be used to assess the impact of lamp parameters on the intensity and resolution of the light it emits.

Novel cathode alloys

The model is a tool that can be used in the development of new cathode materials, particularly multi-element ones, which is the ultimate goal of the research. At the present time, although many cathodes are composed of more than one element and, in principle, emit the line spectrum of each component, it is common to utilize the emission lines of only one element, for which the cathode composition has been optimized.

Making a serviceable multi-element cathode is not easy. If the desired elements readily form a machinable, conducting alloy that is easy to cast, the cathode can be made by consulting standard phase diagrams and its composition optimized empirically by testing the sensitivity (lack of evident self-absorption) of lamp cathodes with different compositions. But few elements are so accommodating. In the absence of a suitable alloy, lamp designers must instead devise novel metal-metal composites and multiphase alloys, guided by educated guesses about the likely structure of the multi-element phase diagram and general knowledge of alloy chemistry.

The kind of understanding and basic research that Monash is well suited to provide demonstrate the benefits of university input. Two alloys already developed during the research project are now entering commercial production, and a third is under evaluation. These alloys will be incorporated in Varian's new range of boosted-output UltrAA lamps, to be released this year.

Derek Oliver is completing his Ph.D. thesis in the Department of Physics at Monash University (Clayton, Victoria), under the supervision of associate professor Trevor Finlayson. Jack Sullivan, a consultant at Varian Australia Pty. Ltd. (Melbourne, Australia) is the industrial supervisor for the project.