concentrations of constituents in a sample, but it has little ability to identify unknowns. Practitioners of another chromatographic technique, gas chromatography, have remedied this deficiency admirably by mating gas chromatographs with mass spectrometers or infrared spectrophotometers in the form of GC/MS and GC/IR instruments. This year at the Pittsburgh Conference, Hewlett-Packard followed up on a hint dropped at last year's meeting by introducing a liquid chromatography/mass spectrometry interface. The Finnigan Corporation actually was first out of the gate with an LC/MS interface; it exhibited its device for the first time last year. A third company, VG Micromass, offered an LC/MS interface but did not exhibit one.

One problem that has prevented effective LC/MS coupling in the past is finding a way to introduce the liquid effluent from an HPLC into the vacuum of a mass spectrometer ionization source. The technique adopted by Hewlett-Packard was adapted from a method developed by Michael Baldwin and Frederick McLafferty of Cornell University in 1973. The interface consists of a long tubular probe whose tip is inserted into an inlet in the mass spectrometer. A needle valve controls the fraction of the HPLC effluent that flows through the probe into a chemical ionization chamber. Solvents in the effluent are used to form the reactant ions necessary for the chemical ionization of the sample constituents. A liquid nitrogen-cooled copper cold trap acts as a cryopump to maintain vacuum in the face of the large pressure increase that would otherwise occur upon vaporization of the liquid effluent.

The interface is an option for the company's top-of-the-line HP 5985B GC/MS system and can be used with any HPLC system meeting certain flow and flow stability requirements. Cost of the device is about $11,500 on a new GC/MS and $20,000 as a retrofit on instruments in the field.

The approach taken by Finnigan and VG Micromass to solve the vacuum/liquid problem is to use a moving belt. Effluent from the HPLC is dropped on the belt, which passes through an infrared heating chamber to remove the solvent. The belt then moves through a two-stage pumping chamber before depositing the sample into either an electron or chemical ionization source, where it is flash heated. The technique is adopted from that described in 1976 by Bill McFadden, H. L. Schwartz, and Stanley Evans of Finnigan. Advantages claimed for the moving belt technique are the ability to take both electron and chemical ionization spectra. Moreover, according to G. L. Kearns of VG Micromass, the moving belt interface can be mated with both high-voltage magnetic and lower-voltage quadrupole mass spectrometers. Finally, the moving belt approach permits processing 30 to 40 samples per hour, as compared to perhaps 3 to 4 with the Hewlett-Packard method.

The Finnigan and VG Micromass LC/MS interfaces, which will operate with most commercial HPLC instruments, are priced at $19,000 and $30,000 respectively.—ARTHUR L. ROBINSON

# Portable Elemental Analyzers Pushed

"It's alright to gamble in Atlantic City but not in the steel yard" might have been the motto of several manufacturers exhibiting optical emission and x-ray fluorescence elemental analyzers at the Pittsburgh Conference. It is not exactly a comforting thought, but steel companies, for example, do not always know what alloys are being stored where in their yards. One alloy may look pretty much like another, but if the wrong alloy were shipped by mistake, and the error went undetected, the results could be catastrophic.

The traditional method of sorting alloys is the spark test in which an experienced metallurgist visually notes the characteristics of sparks (the trajectories of the sparks, for example) emitted when the metal is struck by a high-speed grinding wheel to identify the alloy. Folklore has it that these people were so valuable that they demanded and got high salaries and valuable fringe benefits, just as today's superstars of sports do. If rather subjective, the visual spark test method can be expensive. One steel company needed 300 man-hours to sort out 12,000 pieces that had become mixed up.

Spark emission spectrometers make the testing faster and more quantitative. While such instruments are well known in the various metals industries, what is new are small, portable instruments that can be dragged around salvage, shipping, and storage yards for onsite testing without the need to saw off samples for removal to a laboratory spectrometer. The penalty for portability is that the sensitivity and resolution possible with large and expensive spectrometers is missing. Nonetheless, the capabilities of the portable instruments are quite adequate for alloy sorting and identification. And the price (about $40,000) is small enough that the instruments pay for themselves in a few months to a year, manufacturers claim.

Three portable spark emission spectrometers were shown at the Pittsburgh Conference by Applied Research Laboratories (a division of Bausch & Lomb), the Baird Corporation, and Technics. Baird's machine was shown last year; the other two were exhibited for the first time. All three work in much the same way. A spark source located in a pistol-like probe vaporizes a small quantity of metal at the surface of the sample being analyzed. Atomic emission lines from the vaporized metal are collected by a light pipe in the probe and channeled through a 10-meter-long optical fiber cable to a lens which focuses the light on the primary slit of a direct reading spectrometer. Direct reading means that several exit or secondary slits are located at fixed positions corresponding to wavelengths emitted by the elements to be detected. A diffraction grating disperses the incoming light so that any lines belonging to the elements to be detected are directed toward the proper secondary slit. In this way several elements can be detected simultaneously.

A reference standard is needed to tell the instrument the composition of the alloy being sought, and dials on the front panel of the spectrometer are used to set the range of allowed variations in the percentage of each element. Accept (green) and reject (red) lights on the probe flash on to indicate if the sample tested has the right composition. Testing takes 10 seconds or less. ARL's machine (Quantotest 36000—$36,000) can analyze up to 10 elements. To add new elements requires exchanging the spectrometer optics, which can be done in 10 minutes. Baird's instrument (Spectromobile—$40,000) has a spectrometer with 24 exit slits, but only 5 of these can be monitored at one time. Similarly, Technics'

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portable spectrometer (Spectrotest—$40,000) can test for any 8 of 24 elements simultaneously. The lightest instrument (ARL's) weighs 265 pounds, while the heaviest (Baird's) weighs 395 pounds. All are mounted on wheeled carts.

An alternative to optical emission spectroscopy is x-ray fluorescence analysis. As with optical instruments, large x-ray fluorescence spectrometers are in widespread use in the metals industries for monitoring the composition of alloys in various stages of production. But smaller, less expensive, portable (or semiportable) machines are available that can be used for metal sorting and alloy identification in storage yards or in the field. One advantage of x-ray machines is that they can analyze a wider variety of materials than the optical instruments, including powders, liquids, and samples with highly irregular surfaces. But a disadvantage is that the truly portable instruments are capable of analyzing for only one element at a time and thus are much slower at sorting out alloys with complex compositions.

The grandfather of portable x-ray fluorescence analyzers is made by the Pitchford Scientific Instruments Division of the Hankison Corporation. The Portaspec was introduced 14 years ago. It consists of a tungsten x-ray tube, a lithium fluoride wavelength dispersive analyzing crystal, and a proportional counter detector, all in a probe unit that weighs 18 pounds. A separate power supply unit weighs 55 pounds and is connected to the probe by a 9-foot-long cable. The Portaspec can detect the presence of elements from titanium (atomic number 26) through uranium (atomic number 92) in the periodic table. A meter on the probe unit indicates the intensity of x-ray fluorescence detected and thereby the concentration of each element. Its price is $12,000.

If 73 pounds is too much to carry around, an even more portable x-ray fluorescence analyzer that weighs only 19 pounds was introduced at the Pittsburgh Conference by Columbia Scientific Industries Corp. It can be operated on batteries alone for up to 12 hours. The CSI 740 Compact Materials Analyzer does not use an x-ray tube but a radioactive isotope as the x-ray source, thus overcoming the need for a large, high-voltage power supply. Iron-53, curium-244, cadmium-109, and americium-241 are examples of isotope x-ray sources.

The system uses a gas-filled proportional counter detector operated in an energy dispersive mode, so that several elements can be determined from one exposure. Although the instrument has a microcomputer to store calibration data for up to 32 elements, the microcomputer is only able to process data in groups of four elements. Moreover, there is only one display panel, so all results must be shown sequentially. There are three probe units, which between them cover the elements of the periodic table from aluminum through uranium for liquid, powder, and solid samples. The CSI 740 costs $19,000 with one probe and just over $20,000 with all three probes.

For those who need to determine the concentrations of only one to three elements, the PGT-100 from Princeton Gamma-Tech may do the trick. Rather than a microcomputer, the instrument has analog electronics that are tuned at the factory to process x-ray fluorescence pulses specific to the selected elements. A triple display panel shows the concentrations of all three elements simultaneously. With a hand-held probe, the PGT-100 weighs 40 pounds and its price is about $10,000.

For more analytical capability, the chemist must give up portability. The most sophisticated x-ray fluorescence analyzers that might still be used for alloy sorting and identification, shown by the Kevex Corporation and Princeton Gamma-Tech at the Pittsburgh Conference, might be described at best as semiportable. The Kevex Analyst 6600, for example, has an optional hand-held, liquid nitrogen–cooled, lithium-drifted silicon detector, but the x-ray tube housing and the electronics unit have to be fixed in place, though they need not be in the same location.

A microcomputer in the Kevex instrument gives it several capabilities. It can do quantitative analysis for up to 18 elements in unknown samples; it can identify up to 170 alloys whose compositions are stored in memory; and it can accept or reject unknowns on the basis of the percentages of up to 18 elements in a given alloy. Operation is simple; instructions on a cathode-ray tube (CRT) screen prompt the operator. Results of all modes of analysis are also displayed on the screen. Time for one run can be as short as 5 seconds. First exhibited at the Pittsburgh Conference last year, the Analyst 6600 costs about $50,000.

Princeton Gamma-Tech's PGT-800, introduced this year as a competitor to the Analyst 6600, costs $40,000.

—Arthur L. Robinson

# IC Versatility Promotes Competition

Ion Chromatography is a rapid and sensitive technique for separating and analyzing complex solutions of ionic species. The technique is only 5 years old, but already it is the method of choice for analyzing a variety of organic and inorganic ions in wastewaters, process waters, air samples, biological fluids, food products, and a large number of other materials. It has become so popular, in fact, that some companies are now trying to find ways to achieve comparable analytical success without infringing on the basic patents that cover it.

Ion Chromatography (IC) is a variant of conventional ion exchange chromatography. In ion exchange, a polymeric resin bearing either anionic or cationic functional groups is enclosed in a column and used to separate ions of the opposite charge. A cation resin, for example, bears sulfonate moieties whose attraction for cations in the sample is proportional to the acidity of the cations. When the column is eluted with an acidic electrolyte, the cations that are bound most weakly are eluted first, those bound most strongly are eluted last, and the others are eluted somewhere in between.

Ion exchange is very effective for separating most types of ions—provided the sample can be detected as it leaves the column. Most organic ions and some complex inorganics can be detected spectrophotometrically or by changes in the refractive index of the eluent, but not most simple ionic species, such as halides and metal ions. Ideally, such ions could be detected most efficiently by measuring their electrical conductivity, but the large conductivity of the eluent generally overwhelms any signal arising from the sample. IC provides a way sharply to reduce this background conductivity.

IC was first reported in 1975 by Hamish Small, Timothy S. Stevens, and William C. Bauman of the Dow Chemical Company. They developed both a new separator resin and a unique detection system. Conventional ion exchange res-