

of HPLC at the conference. The five cassettes in the series cover theory, the different types of equipment available, optimization of separations, analysis of results, and other topics. Also available are a manual and an instructor's guide. The complete course costs \$1250. A companion course on gas chromatography is expected to be available within the year.

The cassettes oriented toward specific instruments are relatively expensive compared to commercial cassettes. Finigan's cost \$250 apiece, Varian's are \$195, and EM's are \$150. The cassettes are not included in the purchase price of the instruments in the United States, companies say, because price competition is too great. In the future, says DeJongh, they will probably be included

in the package for sales abroad because service is a greater problem there. Shipments to developing countries, he adds, may even include a videocassette player to induce the user to perform more preventive maintenance. "For customers in relatively isolated areas," he concludes, "anything they can do to prevent the need for service calls is a benefit for both them and us."—**THOMAS H. MAUGH II**

A New Dimension in Gas Chromatography

Gas chromatography (GC) has undergone a renaissance during the last 5 years as a result of the introduction of reliable capillary techniques. These techniques make gas chromatograms highly reproducible—to the point where retention times can be used for analysis of unknowns—and have permitted unprecedented separations. Nonetheless, the separation of complex mixtures is still often a problem because two or more components of such a mixture will often have similar retention times on a given column. Most of these separation problems can be solved with a new technique known as multidimensional GC or MDGC.

In essence, MDGC involves the use of two or more separate GC columns for a given separation; ideally, each column will reside in its own oven so that its temperature can be controlled independently. Most often, the second column will have a markedly different polarity from that of the first column to enhance separation of components that are not separated cleanly in the first column.

The two columns can be connected serially to provide increased separations, but the most common application is column switching for heartcutting. In this case, most of the sample eluted from the first column goes directly to a detector, but an inadequately resolved fraction is shunted to the second column for more complete separation. This approach is especially useful for the analysis of a trace component in a complex mixture or a minor component eluting immediately after a major component. MDGC can thus sharply reduce the time required for analysis of specific components in complex mixtures and can often provide separations that are impossible with other techniques.

MDGC was originally conceived in the mid-1960's by David Deans of ICI Ltd. in England, but it did not receive much attention until the development of capil-

lary columns in the late 1970's. The technique has subsequently been used to a significant extent in Europe, but is only now generating interest in the United States. Three new instruments for general MDGC were displayed for the first time in the United States at this year's Pittsburgh Conference.

The key to MDGC is the use of special devices to perform the shunting. Shunting can be achieved with a conventional mechanical valve, but such valves typically are large, which means they are slow to equilibrate in the oven, and often have large dead volumes, which degrade resolution. Only recently have acceptably small valves been developed.

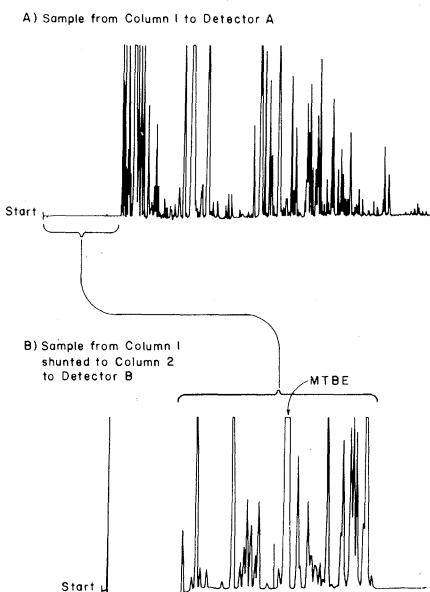
Deans developed what is known as a valveless coupling piece, a so-called "live"-switching device. The coupling piece has a small mass and minimal dead volume; most important, the direction of

gas flow is controlled by small variations in gas pressure rather than by mechanical devices. The use of a coupling piece gives more sensitive and precise control of heartcutting and other shunting operations and is most valuable when shunting must be performed several times during a particular run. Counterbalancing this flexibility is its increased complexity compared to a mechanical valve.

A typical application of heartcutting was described at the conference by Susan Sonchik and John Walker of IBM Instruments, Inc. (Danbury, Connecticut). They demonstrated the analysis of the octane enhancer methyl-*t*-butyl ether (MTBE) in gasoline. Gasoline is a very complex mixture of hydrocarbons, and MTBE typically elutes in the same region as many short-branched hydrocarbons. To analyze for MTBE, all components of the mixture that elute before *n*-heptane are shunted to the second column, which is eluted under different conditions to enhance separation (see figure). In this case, the shunted fraction occurs at the beginning of the chromatogram, but it could occur anywhere during the separation.

The remainder of the gasoline components normally flow directly to a detector. Alternatively, analysis time can be shortened by a technique called backflushing, in which the flow of carrier gas through the first column is reversed to remove the heavier components from the column more quickly (since they will be closer to the injection port than to the detector). Backflushing is especially useful for components that would elute only very slowly under normal conditions.

The new instruments displayed at the conference are the GC/9630 from IBM, the Sichromat 2, manufactured by Siemens AG of West Germany and distributed in the United States by ES Industries (Marlton, New Jersey), and the Series 500 from Hach Company (Loveland, Colorado). All three are fully auto-



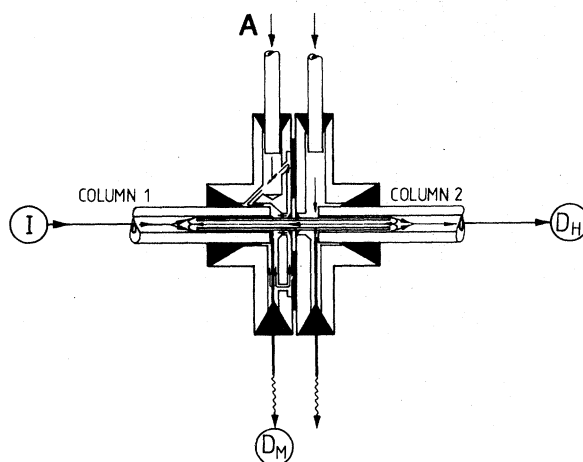
Heartcutting

*Determination of methyl-*t*-butyl ether (MTBE) in gasoline by heartcutting. To analyze MTBE, all components of the first column that elute before *n*-heptane are shunted to the second column. [Source: IBM]*

mated. The GC/9630 and the Sichromat 2 have two ovens whose temperatures can be independently controlled; the Series 500 has a third oven for accessories. The Sichromat comes with a coupling piece and the Series 500 comes with a mechanical valve, but the GC/9630 can be ordered with either a coupling piece or a mechanical valve. An IBM spokesman contends that the valve is satisfactory for more than 80 percent of potential applications.

All three GC's can be equipped with two sets of injectors and detectors so that the two columns can be operated in parallel (when, for example, it is desirable to separate a mixture under two sets of conditions to provide a definitive identification of components) or completely independently. The price of the GC/9630 ranges from \$14,000 to \$22,000, depending on the options selected; the base price of the Sichromat 2 fully equipped for MDGC is slightly under \$30,000; the price of the Series 500 ranges from \$15,000 to \$25,000.

MDGC can also be carried out with three or four columns. E. G. Boeren of Packard Instrument B.V. (Delft, the Netherlands), for example, described a three-column system for analyzing paraffins, olefins, naphthenes, and *iso*-aroma-



"Live" switching

The coupling piece devised by David Deans has no moving parts; the direction of flow of eluant gas is controlled by small differences in air pressure. Changes in pressure through inlet A can shift the flow of eluant from column 1 either to a detector (D_M) or to column 2 and its detector (D_H) [Source: Siemens AG]

tics in naphtha. The naphtha is first passed through a polar precolumn that separates it into two groups, one containing aromatics and the second containing saturated hydrocarbons and olefins.

The second fraction is passed through an Olefin Trap that captures all olefins containing more than five carbons. The saturated hydrocarbons pass through the trap and are carried to a column in a heated zone above the oven where they are separated according to number of carbons. The flow of carrier gas through the trap is then reversed and the temperature raised to release the olefins;

they then pass through a reactor which hydrogenates them for separation on the column in the heated zone. Meanwhile, the aromatics are separated by number of carbons on another column in the oven. A GC incorporating all these features and known as the model 412A PiANO Analyzer was displayed at the conference by Packard Instrument Company (Downers Grove, Illinois). This device is designed for petroleum refiners and for ink and paint manufacturers who must know the precise composition of naphtha, and sells for just under \$35,000.

—THOMAS H. MAUGH II

Ion Beams for Compositional Analysis

Many analytical techniques can be classified according to the kind of particles (photons, electrons, atoms, ions, and so on) that irradiate an unknown and the kind of particles that emerge from the sample to be detected. Secondary ion mass spectrometry (SIMS) is a highly sensitive, surface-specific technique that makes use of ions (although not the same ones) in both roles. This year's Pittsburgh Conference saw the introduction of two other ways to use ions to quantitatively determine concentrations of elements in solid samples, as well as continued progress in SIMS instrumentation itself.

In SIMS, the bombarding or primary ion beam, which can be any ion (argon, oxygen, nitrogen, and cesium are common) has an energy of a few thousand electron volts. Collisions with atoms near the surface soon bring the ions to a halt, dislodging in the process a conglomeration of neutral atoms and negatively and positively charged ions that fly away from the surface. As many of the low-energy secondary ions as possible

are collected and transported to a mass spectrometer (almost always a quadrupole analyzer).

Advantages of SIMS include a high sensitivity (in the parts-per-billion range for some elements), a fairly high spatial resolution (down to 1 micrometer or so), and an ability to do depth profiling; that is, obtain concentrations as a function of depth below the initial surface, when the surface layers are one after another sputtered away by the primary ion beam. The main disadvantage is a tremendous variation (a factor of 10,000 or more) in the efficiency of producing secondary ions, according to the identity of the ion, its neighbors in the solid sample prior to being sputtered, and the primary ion species. This variation makes SIMS difficult, although not impossible, to quantify.

Rutherford backscattering (RBS) is, to physicists at least, an ancient technique that has now been neatly packaged by the General Ionex Corporation (Newburyport, Massachusetts) and presented for the first time to Pittsburgh Confer-

ence attendees in the form of the model 4175 Rutherford Backscattering Surface Analyzer.

RBS by no means replaces SIMS but instead is complementary. The sensitivity is about 10,000 times poorer than that of SIMS, and there are for the moment no focused-beam, scanning systems to provide a high spatial resolution. However, the technique is absolutely quantitative and there is no need whatsoever for standards. Moreover, RBS is nondestructive. It permits quantitative depth profiling of all elements from lithium through uranium without the need to sputter away the surface.

The quantitativeness comes from the fact that the physical principle of RBS is not much more complicated than that in the freshman physics laboratory experiments studying elastic collisions between steel balls. A beam of doubly ionized helium ions of energy 2 million electron volts (MeV) enters the surface. When an ion strikes an atom in the sample head on, the ion reverses its path and comes back out. The energy of the