The Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy is the largest meeting devoted to chemical instrumentation in the United States, and probably the largest in the world. From 1 to 5 March, more than 7500 scientists gathered at the Cleveland Convention Center to listen to some 497 papers and to visit the booths of 324 instrument manufacturers and distributors. Another 3000 visited only the instrument exhibition. Because so many manufacturers exhibit their established instruments and introduce new ones at the Pittsburgh Conference (which is so named because it is sponsored by the Society for Analytical Chemists of Pittsburgh and the Spectroscopy Society of Pittsburgh even though it has been held in Cleveland for the past 9 years), it is an ideal place to examine trends in instrumentation and, thereby, current trends in chemistry. Many of those trends are discussed in the following stories. Other trends include the growing number of women participants. As recently as 5 years ago, there was only one woman among the manufacturer's representatives at the meeting. No figures are available this year, but women probably accounted for at least 5 percent of the sales force. Also the number of professional women visiting the exhibition was much greater than ever before. Another important trend was the enthusiasm of the visitors to the exhibition. Many of them seemed to have money in their pockets and to be ready to spend it, an indication that the instrument business—and, therefore, the physical sciences—will enjoy a year of healthy growth.

Fourier Transform: The Revolution Comes to Infrared

In conventional infrared spectroscopy, a great deal of effort and expense is expended in the collection of some 4000 data points for each spectrum, but only one or two dozen of the points are used to define the location and intensity of spectral peaks. More than 99 percent of the collected data, according to Tomas Hirschfeld of Block Engineering, Cambridge, is discarded. Most of these data can be recovered and used to provide additional information, however, with the technique of Fourier transform infrared (FT-IR) spectroscopy. Just as Fourier transform techniques have revolutionized nuclear magnetic resonance spectroscopy—making feasible the routine study of very small samples and of nuclei other than hydrogen—FT-IR promises to effect a small revolution in the use of infrared spectroscopy.

The increased sensitivity, versatility, and accuracy of FT-IR spectrometers compared to conventional instruments result from the combined use of a Michelson interferometer, a minicomputer, and a laser wavelength reference. The interferometer is, in essence, a mirror device that converts infrared (or other electromagnetic) radiation into signals in the audio range (an interferogram) in much the same fashion as a heterodyne radio converts electromagnetic radiation into sound. Its advantage is that the entire spectrum of interest is recorded simultaneously, rather than point by point as in a conventional dispersion spectrometer. Furthermore, the incident light is admitted to the interferometer through a large aperture rather than through the conventional narrow slit, thereby allowing a greater amount of light to reach the detector, whose sensitivity is the limiting factor in most conventional instruments.

The incident spectrum can then be reconstructed from the interferogram by a complex, though rapid, mathematical manipulation—the Cooley-Tukey algorithm—that is accomplished by the minicomputer. The minicomputer also provides the capability for averaging large numbers of spectrums to increase the signal-to-noise ratio (and thus the sensitivity) and for manipulating the spectrums in ways discussed below. High accuracy of wavelength determination, which permits both high reproducibility and addition and subtraction of spectrums, is achieved through use of a highly stable laser to provide an internal wavelength reference.

These and other features make it possible for an FT-IR spectrometer to perform tasks that are impossible, or nearly so, with conventional instruments. One of the most dramatic of these is the coupling of a gas chromatograph with an FT-IR spectrometer for the separation and identification of small amounts of materials. Conventional infrared spectrometers require too long a scan period and too large a sample for this to be feasible. New instruments displayed at the Pittsburgh Conference by Digilab, Inc., of Cambridge can produce spectrums "on the fly" from gas chromatograph samples smaller than 200 nanograms. This capability for the first time makes the instrument as sensitive as a mass spectrometer combined with a gas chromatograph.

Another capability is the production of spectrums for individual components of a mixture; in a sense, this is a chemical separation performed by a computer. One example explored by J. L. Koenig and his associates at Case Western Reserve University is the analysis of semicrystalline polymer systems. Polyethylene terephthalate, for instance, is composed of crystalline and amorphous phases that are difficult to obtain individually. Koenig has shown that it is possible to obtain a spectrum of each phase by FT-IR spectroscopy. This is accomplished by obtaining a spectrum of a sample, annealing the sample to increase the proportion of crystalline material, and then obtaining a second spectrum. Manipulation of the spectrums with the minicomputer then produces a spectrum of each phase. Similar feats can be accomplished with mixtures of as many as five components, Hirschfeld says, through use of any technique that will produce a change in the proportions of the components.

It is also possible to identify thin organic films on the surface of various materials. It is already routine to obtain spectrums of such films on the surfaces of metals, and it is also possible to obtain them when the film is on the surface of a material that absorbs in the infrared. One example shown by Robert J. Jakobsen of Battelle Columbus Laboratories is the study of the binding of various blood plasma proteins to polymers and surface-treated polymers of the types used for implants. It is relatively easy to obtain a spectrum of the proteins bound to the plastic; by subtracting the previously obtained spectrum of the plastic, one obtains a spectrum of the proteins. And if spectrums of the pure proteins are available, these can also be subtracted to reveal the spectrums of those functional groups that participate in the binding and are thus slightly altered.

Similar results can be obtained with other types of materials. James Brasch of the University of Maryland has shown that it is possible to study fire retardants and other agents on the surface of textile fibers. R. R. Willey of the Willey Corporation, Melbourne, Florida, has demonstrated the capability of FT-IR to obtain spectrums of coatings and other treatment agents on the surface of papers.

And Kenneth L. Kizer of Digilab has found that it is possible to study trace amounts of substances—such as pollutants in water and additives in polymers—in most liquids and in many solids.

Another interesting capability of FT-IR permits measurement of the kinetics of high-speed reactions. Spectrums can be obtained in a few microseconds, so that transient intermediates can be identified by their spectrums and reaction rates can be calculated. William G. Fately of Kansas State University, for instance, cites the demonstration that FT-IR can be used to identify a ketene intermediate in the flash photolysis of acetone, even though the reaction is over in about 100 microseconds. Such reactions can be followed with other techniques, but FT-IR is unique in that it provides a complete spectrum of the substrates and intermediates at each stage of the reaction.

Many of the scientists who have initiated investigations of FT-IR have had to use instrument systems they have put together themselves. Now, however, a variety of high quality instruments are marketed commercially. Four companies were promoting them at the Pittsburgh Conference: Digilab; Willey Corporation; Nicolet Instrument Corporation of Madison, Wisconsin; and Coderg S.A. of Clichy, France. The versatility of FT-IR, of course, does not come cheap. Prices range from about $40,000 for the stripped down models to $100,000 or more for top of the line instruments—a far cry from the $3000 price of a small conventional infrared spectrometer. But for many applications that are possible only with FT-IR, those prices are certainly not expensive.

—THOMAS H. MAUGH II

The Intelligent Instrument: Coming, but Not Yet Here

Envisage a wide array of laboratory instruments under the control of their own internal computers, automatically calibrating themselves, taking and analyzing data from large numbers of samples, repeating procedures that gave questionable results, and warning operators or other machines when certain conditions are exceeded. It is a fascinating prospect, the age of the "smart" instrument, but it is not here yet. Indeed, despite continuing rapid developments in the capabilities and economies of microprocessors—the computer on a chip that makes it all possible—there is every indication that it will be at least several years before many such instruments, even in more modest versions, will be available. Still, a few relatively expensive instruments with impressive microcomputer control and data reduction capabilities are being sold now. More are being introduced with either digital controls that are compatible with computer operation or microprocessors that perform limited data-handling tasks, both roadmarks on the way to a fully integrated instrument. Based on the exhibition at the Pittsburgh Conference, here is an impression of where the action is and what the next few years might hold.

Gas chromatographs are leading the way to more computing power for data reduction and instrument control. Four manufacturers now offer an integrator, built around a microprocessor, for use with the analytical instrument. These essentially separate, add-on devices convert chromatography peaks into concentrations and do similar data-handling chores more rapidly and more accurately than was previously possible. Since most such integrators can be used with any chromatograph, they provide a means of upgrading older instruments. Examples include units manufactured by Spectra-Physics of Santa Clara, California, Columbia Scientific of Austin, Texas, and Hewlett-Packard of Palo Alto, California, and are priced from $3000 to $5000.

The future, however, would seem to belong to microcomputers that are, in a sense, built into the chromatographs, so that they interact with the instrument and provide the programmable controls for essentially automatic operation. The most ambitious experiment in this direction is a completely automated chromatograph introduced last year by Hewlett-Packard. Its design is one in which the microprocessor is integral to the instrument and controls every event in the chromatograph, from turning on the oven to injecting samples. The $10,000 unit (an automatic sampler is extra) prints out and plots a complete analytical report. Additional flexibility in control of the instrument was added this year, making it possible for the unit to change operating procedures or data-handling methods during a run—sample by sample, if necessary. To make use of such flexibility, however, a host of run parameters, calibration and analysis specifications, and control options must be chosen in advance. To avoid repeating this extensive programming, the parameters can be saved on a magnetic card and reentered from it. Despite its cost, the instrument is reportedly selling well, which may be evidence that the cost of laboratory technicians and operator errors is greater still.

A somewhat different approach is evident in a new chromatograph made by Varian of Walnut Creek, California, that can be operated either alone or, coupled with a separate microprocessor unit, as an automated system. Here the microcomputer is used more selectively than in the Hewlett-Packard unit to control or to check most chromatograph functions, as well as to handle data reduction; it can also be operated separately as an integrator with other chromatographs. This interactive but separate approach, Varian spokesmen claim, makes it easier for the user to do such things as locating and repairing simple malfunctions without manufacturer assistance. Both the Hewlett-Packard unit and the comparably priced Varian instrument can transmit data directly to other computers and will sound alarms if certain preset parameter limits are exceeded. They are complicated instruments—so much so that Hewlett-Packard is thinking of establishing courses for operators. All manufacturers contacted expect the computing power and the control logic of chromatographs to increase rapidly.

Atomic absorption spectrometry is another area where microprocessors are beginning to appear. A unit from Perkin-Elmer, Norwalk, Connecticut, incorporates one, but uses it for data reduction only; the instrument itself operates with analog controls. A new instrument from Jarrell-Ash, Waltham, Massachusetts, is completely digital and uses its microprocessor as the control center for the unit—automatically recalibrating itself, finding a designated wavelength, and searching for the most intense absorption peak in that spectral neighborhood. As a result, the instrument is said to be relatively simple to use, even for inexperienced operators, and it can be run remotely from a teletype terminal. At a cost of nearly $11,000, it is more expensive than other atomic absorption spectrometers.

Spokesmen for Jarrell-Ash say the greatest difficulty in developing the instrument was programming the micro-