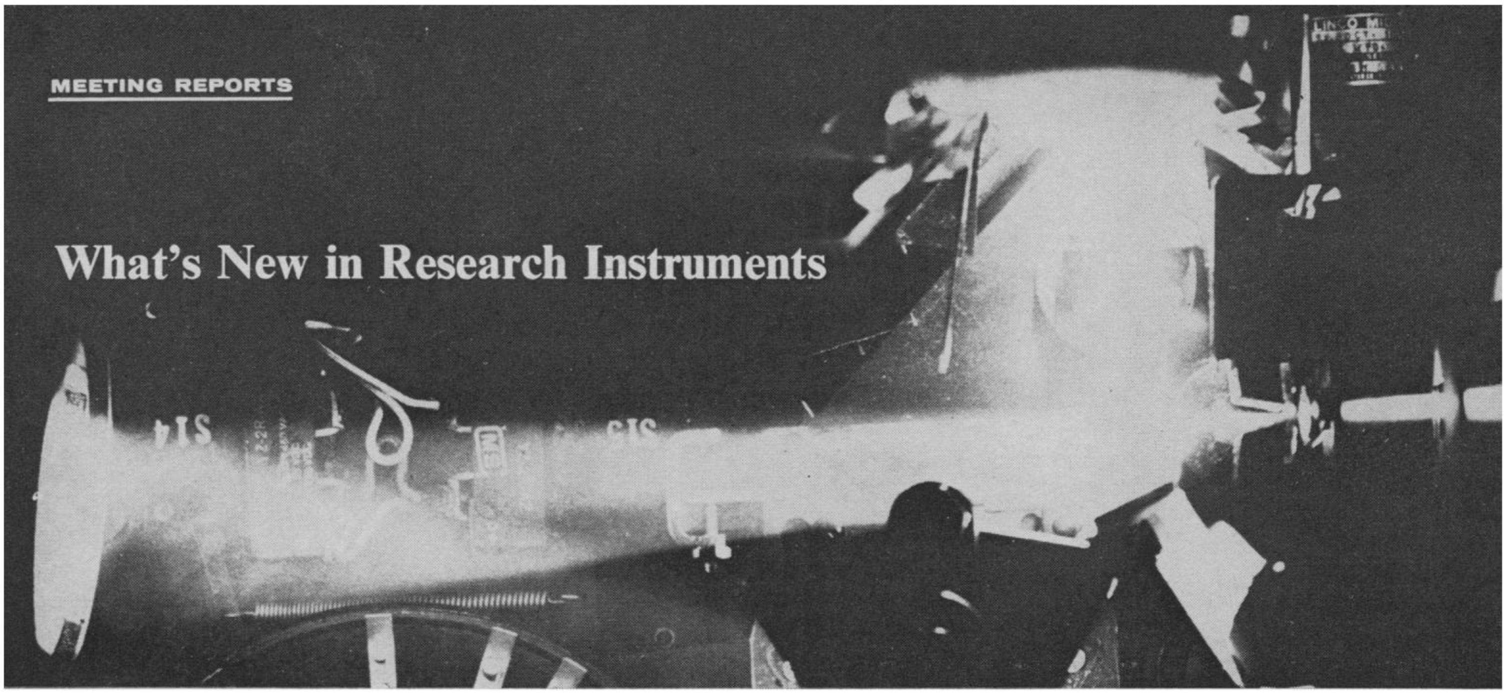
What's New in Research Instruments



Perkin-Elmer Corp. photo shows path traversed by infrared energy in infrared spectrometer.

It takes more nowadays than a nodding acquaintance with quantum mechanics and some of the new mathematics to introduce new physical methods of chemical analysis. Perhaps the main thing it takes is a bankroll. Instruments which could be ordered at the recent Pittsburgh conference on analytic chemistry and spectroscopy (2–6 March) range from a price of around $6000 for the rugged, tabletop infrared spectrophotometer that has become the routine industrial tool for quantitative analysis of chemical unknowns to a price of $1 million for the equipment for neutron activation analysis, including a nuclear reactor (the University of Mexico recently bought the works from General Dynamics' General Atomic Division). Researchers interested in acquiring the new interferometer shown for far-infrared measurements would have also to think of the cost of computer time; many a university would be out looking for an arrangement like that which links 45 New England colleges and universities to the $3-million IBM 7094 computer at Massachusetts Institute of Technology (*1*).

The instrument industry has followed researchers into almost every frequency of the electromagnetic spectrum to produce energy probes that are, on the one hand, greatly extending analytic possibilities and, on the other, revealing new facts about the fundamental forces that determine molecular structure and reaction mechanisms. To the instrument makers' show to see what's new came some 5000 scientists of a great variety of interests—from such security-restricted laboratories as Lawrence Radiation Laboratory, where no salesman can call; from the international petroleum industry, where representatives carefully avoid showing a direct interest that might tell competitors what they are up to; from the FBI, whose scientists now sometimes take minute samples to reactor sites where neutron activation analysis may show that a suspect has fired a gun (*2*).

Mössbauer Effect Analyzer

In scientific principle, the newest instrument at the show was the Mössbauer Effect Analyzer, which Nuclear Science & Engineering Corporation began to produce last January. Designed initially as a demonstration instrument for science teaching, the analyzer is based on the principle discovered in 1958 by Nobel laureate R. L. Mössbauer: Certain radioactive nuclei embedded in solids can emit and absorb low-energy gamma rays without recoil.

Since this discovery many researchers have built instruments which, like the one Mössbauer developed initially, make use of the Doppler effect for detecting resonance absorption of the emitted gamma rays. When a mechanical motion of source or absorber is used to change the energy of the gamma rays, maximum absorption will be found to occur at specific velocities for each combination of nucleus and environment examined. Specific chemical ligands of atoms whose nuclei can be studied by this means are revealed as shifts of the basic absorption lines, magnetic interaction by splitting of the lines.

Such research instruments are not easy to build. For example, J. Spijkerman, who is exploring possibilities of the Mössbauer technique at the National Bureau of Standards, said it took a year to build his instrument. This instrument has been designed to achieve the most accurate calibration possible; velocity is measured by using the wavelength of light (via an interferometer) as the yardstick of distance and the Bureau's atomic clock based on the vibration frequency of cesium atoms as the time standard.

Mössbauer effect is a very active field of research; a large number of radionuclides are known to have lifetimes long enough to permit measurement. For some experiments, the technique has the highest resolution so far achieved in the laboratory. Chemists are using Mössbauer effect to study reaction mechanisms too rapid to examine by other means by observing changes in electron densities at the nucleus. Physicists are using the technique to study such fundamentals as

This summary report on new instruments was made possible by the help of each one of the scientists named in it and also by help from Francis P. Byrne of Westinghouse Research Laboratories, general chairman of the 1964 Pittsburgh Conference; Gunther K. Wertheim of Bell Telephone Laboratories; D. R. Lide, H. E. Radford, and Joshua Stern, all of the National Bureau of Standards; and Robert L. Bowman of the National Institutes of Health.

the nature of magnetism and nuclear lifetimes and moments. They have even succeeded in measuring the difference between nuclear radii of ground and first excited states.

At the last international conference on Mössbauer effect, applications were discussed ranging from fundamental studies of this sort to study of how iron is bound in hemoglobin and of vibrations in the small bones of the ear (*3*).

This research has been done with instruments built by the researchers, most of which have been designed for a continuous sweep through the velocity range. The absorption spectrum is built up by scanning the Doppler velocity many times per second over the study range and by synchronous sorting of gamma-ray counts into a multichannel analyzer. Absorption spectra are displayed on an oscilloscope or printed out on tape.

The Nuclear Science & Engineering instrument, first commercial instrument available for making Mössbauer measurements, differs from most of these research instruments in the respect that velocity variations are selected as discrete points over the available range of 0 to 15 mm per second by means of a manually operated dial. Gamma-ray counts at specific velocities are shown numerically on a scalar, and the experimenter uses the data to plot the graph that constitutes the absorption spectrum. Thus the commercial instrument should be particularly useful in student laboratories. The manufacturer also said that it had found wider applications. The instrument is priced at $1770 with accessories.

So specialized is the demand for instrumentation when a new research approach is opening that companies sometimes get a new line started on the basis of a single order. Perkin-Elmer, for example, organized in 1937 to provide an American source for optical goods competitive with those of the few European firms then dominating the field, built its first commercial infrared spectrophotometer in 1944 after designing an infrared optical system at the request of a chemical company working on synthetic rubber.

This year at Pittsburgh a new firm, NMR Specialties Inc., showed a pulsed resonance spectrometer it had designed at the request of Riki Kobayashi at Rice University in Houston. Kobayashi, who is studying the rate of diffusion of certain gases, is interested in pulsed resonance frequency which, in contrast to continuous-wave nuclear magnetic resonance (NMR), changes the spin orientation of nuclei and then permits observation of the relaxation time.

NMR Specialties has also built a nuclear quadrupole resonance spectrometer which provides supplemental information about chemical bonding and crystal structure in solids by replacing the external magnetic field of the conventional NMR instrument by the internal electric field of the crystal lattice. This permits measurement of the interaction of the quadrupole nucleus with the internal electric field gradient.

"Perhaps not more than two dozen men are now working on nuclear quadrupole resonance," Frank Dickson, a former Mellon Institute researcher who is executive vice-president of NMR Specialties, said. "But we believe the availability of an instrument adapted for nuclei of many solids over a wide range of frequencies will greatly increase research interest in this technique."

Among many other new developments to be seen at Pittsburgh were:

► A superconductor magnet with a field intensity of 47,000 gauss and a field uniformity that permits a resolution of $1/10^8$ across a spinning 5-millimeter sample tube (this was shown in operation with a nuclear magnetic resonance demonstration by Varian Associates).

► Substantial size reductions in many lines, no doubt assisted by the electronic miniaturization that has prepared instruments for space travel. [Beckman Instruments, for example, has delivered to Jet Propulsion Laboratories a prototype lunar gas chromatograph of just 14 pounds (6.3 kg) and $8 \times 8 \times 10$ inches ($20 \times 20 \times 25$ cm).]

► A widened range of instrumentation for biochemical analysis.

► Work which Frederick Brech, coinventor of the laser microprobe, says may soon make this instrument a tool for quantitative assays.

Laser Microprobe Advancing Toward Quantitative Assay

The ruby laser microprobe is described by its manufacturer, Jarrell-Ash Company, as the first analytic instrument to make use of a laser beam. Replacing conventional arc or spark excitation, the laser can be used with any emission spectrometer. Focused on the sample through a microscope, the laser beam gives spectra within 1 second by vaporizing solid samples not more than 50 microns in diameter. Elements present in the vapor are raised to emissive energy levels by a cross-excitation system. The minute area and the fact that samples need not be electrically conductive is expected to give the instrument especially wide usefulness in analysis of the biochemistry of living systems.

"We are putting a tool into the hands of organic chemists, biophysicists and medical researchers, nondestructive as well as quantitative, that will make it possible to analyze skin and bone with the same facility and ease that metal is analyzed by conventional spectroscopy," Brech said. "But the laser is demanding a new technology. We need sapphire plates instead of a rotating filter. We must have a microscope lens attachment that will withstand power amounting to 10^{14} watts per square centimeter.

"As yet the laser microprobe does not permit fully quantitative assays, and the reason resides largely in the variation of energy output of the laser itself. Nevertheless, a sizeable advancement in the state of the art is being made, by achieving delivery of the stored energy in the laser in a single giant pulse. This in turn has required the development of improved components, such as high speed motors with shaft wobble of not more than 0.0001 cms."

Stanley D. Rasberry told about work at the National Bureau of Standards with the laser microprobe which indicates that a high-temperature-resistant cement for microscope lens components may solve one of the technological problems.

Some thought the laser microprobe was only the beginning of a new era in spectroscopy. If the great variety of electromagnetic energy probes now available have outdated classical analytical techniques that begin with boiling points and melting points and may extend to degradations requiring weeks of a skilled analyst's time, the possibility of eventual development of tuneable lasers delivering intense energy at frequencies ranging through broad regions of the spectrum suggests that the next decade may see another great change in analytic methods.

The far-infrared region of the electromagnetic spectrum, where frequencies lie just above the microwave sector exploited in radar development and just below the infrared bands now used routinely in analysis of chemical unknowns, is a part of the spectrum only recently opened to investigation. New instruments available for accurate measurement at far-infrared frequencies were shown at the Pittsburgh conference, and new problems are persuading researchers to use them.

In plasma physics, for example, microwave techniques have been heavily used to study the kinetic behavior of electrons that determines plasma temperature. But to study high-density plasmas required for controlled thermonuclear reactions, observations must be extended to the higher frequencies of the far-infrared region. Similarly, study of other types of plasmas—for example, how to shield against the ionic sheath that causes communication breakdown when a spacecraft reenters the earth's atmosphere—requires observations extending into the far infrared.

In solid-state physics, the lattice vibrations of crystals are primarily detected within the range of far-infrared radiation. While the basic experiment with germanium and silicon was carried out at microwave frequencies, in many other materials with semiconductor properties, carrier electron frequencies are in the far-infrared range (*4*). Frequencies of electrons excited in superconductors lie entirely in the far-infrared region (*5*).

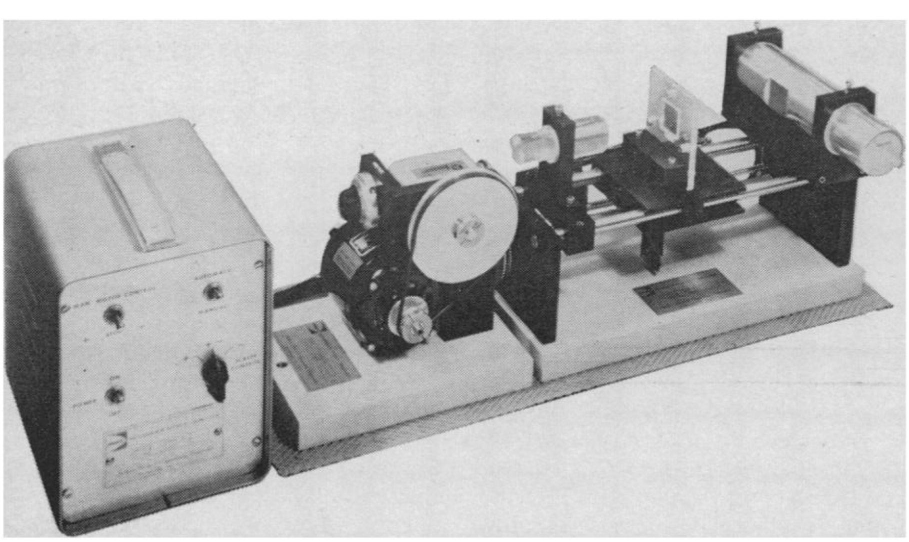
Physicists pursuing the question of how much energy it takes to make or break molecular bonds, as well as the study of other sorts of molecular behavior, have also been probing into the far-infrared, while analytic chemists are finding it possible to make structural determinations of inorganic molecules from far-infrared absorption frequency patterns.

Infrared radiation below 300 cm^{-1} (wavelength longer than 33 microns) is absorbed by organic molecules and converted into internal rotational energy; thus, this rotation can be studied in both the far-infrared and the microwave region. Torque or hindered rotation around single bonds is one of the interesting areas being explored at far-infrared frequencies. W. G. Fateley of the Mellon Institute reported on how he and his associates have been able to measure torsional frequencies for certain C–C bonds and to use these in calculating barriers to internal rotation in some straight-chain compounds and conjugated systems. He has also calculated potential barriers for methyl groups attached to a benzene ring.

Since the radiation absorbed by organic molecules and converted into vibration energy lies in the infrared spectrum from 10,000 cm^{-1} down to 10 cm^{-1}, it is necessary to pursue vibration studies for only a few organic bonds to the far-infrared (usually defined as beginning about 200 cm^{-1}). But some of these low-energy vibrations are extremely important, and the very weak H bond, which sometimes seems to shift between two neighbors, is one of them. Stretching and bending frequencies of alkyl halides also lie in the far-infrared range.

R. J. Jakobsen and J. W. Brasch at Battelle Memorial Institute have been able to measure stretching vibrations for the –O–H . . . O– bond for a series of substituted phenols. For this and other analyses, Jakobsen said they had developed a technique for absorbing liquid samples into molded polyethylene disks; the disks absorb the liquid samples when heated to 110°C. This matrix permitted both solution and low-temperature studies without the disadvantage of obscuring lattice vibrations. The technique, Jakobsen said, greatly facilitates study of weak intermolecular forces such as H bonding. A Perkin-Elmer 301 far-infrared spectrometer was used for this work.

Mössbauer effect analyzer is the first commercial instrument in this field.



Improved Far-Infrared Instruments

The increased interest in the far-infrared region has persuaded Perkin-Elmer to add a grating to increase the range of its 301 spectrophotometer. This was the first commercial instrument available for far-infrared work, and before its advent in 1961, researchers entering this field built their own instruments. The 301 now has a range of 660 to 32 cm^{-1}, uses four gratings, a variety of transmission filters and Reststrahlen plates, and costs $32,500.

Beckman Instruments, Inc. announced a new model far-infrared spectrophotometer, the IR-11, which has a scanning range of from 800 to 33 cm^{-1}. Four gratings and eight filters are provided, and the price is $33,000.

"Recent work in our laboratories has led to the development of a series of highly efficient transmission filters for the far infrared which permits the complete elimination of Reststrahlen filters," Gerald T. Keahl said in a paper on the IR-11. "These filters transmit about 40-70 per cent in their useful ranges with very high rejection of unwanted frequencies. The advances in filtering and convenience of operation make 15-minute scans feasible for this difficult region."

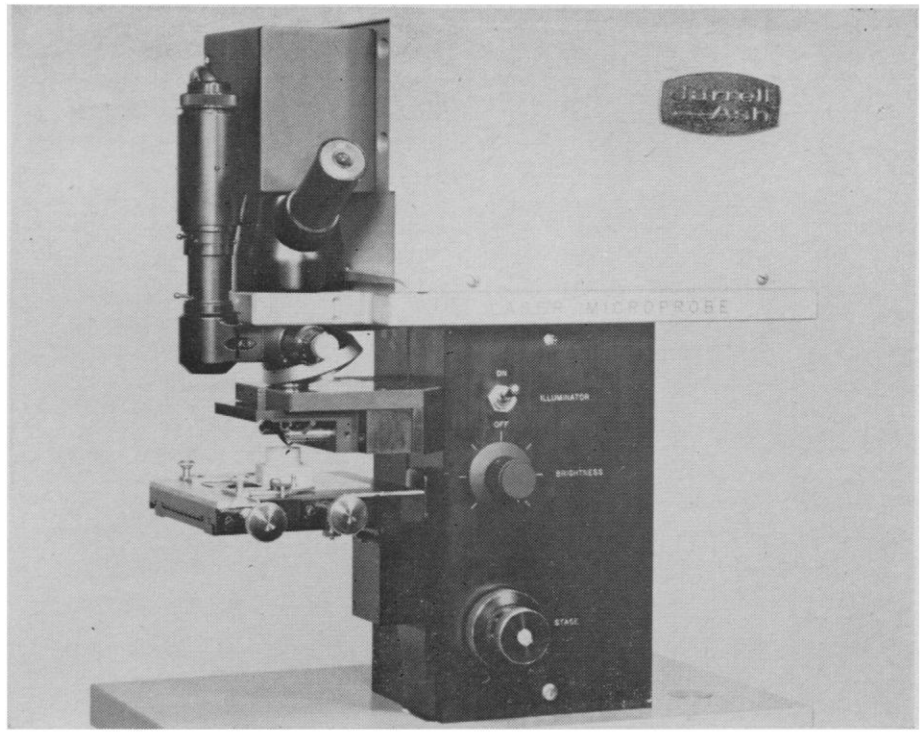
Beckman also showed two other new spectrophotometers with extended ranges in the infrared region. The IR-10 has a range of 400 to 300 cm^{-1}, costs $6800, and is described by the manufacturer as having the "widest wave length range of any instrument in its price class." The IR-12, described as the "first automatic spectrophotometer to cover the 400 to 200 cm^{-1} range," costs $21,000.

The new interferometer being made by a British firm, Research & Industrial Instruments Company, was the star of the far-infrared show, and almost every prober in this region thought of some new thing he would be able to see with it. The interferometer covers a far-infrared range from 500 cm^{-1} down to 10 cm^{-1}, thus reaching wavelengths of 1000 microns, but those who want to try it must have access to a digital computer.

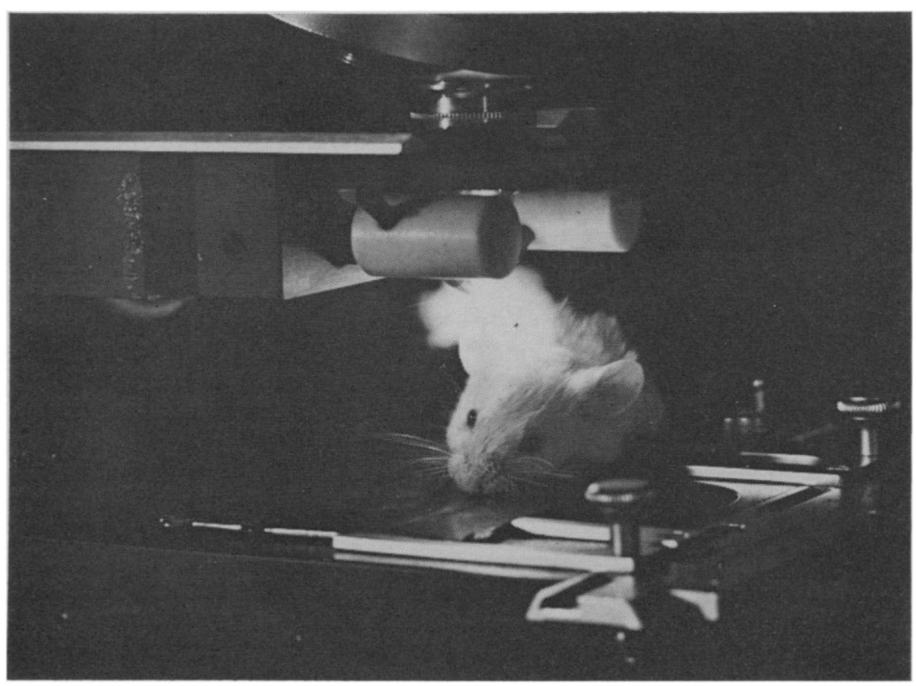
This "Fourier Spectrophotometer" is similar to the prototype designed by Britain's H. A. Gebbie (*6*) and based on the simple optical paths of Michelson's interferometer. In the interferometer an infrared beam is split, traverses two different paths, and then is recombined, the two halves interfering to produce the interferogram. The recombined beam passes through the sample, which modifies the interferogram, and the difference between the modified and the basic interferogram is detected by a Golay cell. The complete instrument (FS-520), which sells for $12,500, includes an analog-to-digital converter and a punch which produces the interferograms in pure binary code, ready for insertion in a general-purpose computer, programmed to convert the digitalized data by means of the Fourier transformation to the familiar absorption spectra.

J. N. A. Ridyard of the manufacturing firm described the new instrument, emphasizing its efficient use of the low energy available in this region. The conventional grating spectrophotometer scans only one spectral element at a time, he said, with the rest of the radiant energy falling on the jaws of the optical slit for most of the experiment. The interferometer gives spectra over a very wide frequency range in a single scan, with no time lost for grating and filter changes.

The instrument is being marketed in the U.S. by Limit Research Corporation, a subsidiary of R.I.I.C. The Atomic Energy Commission laboratory at Los Alamos bought the instrument shown at Pittsburgh; the Research Laboratory of Electronics at Massachusetts Institute of Technology has just received delivery of another.



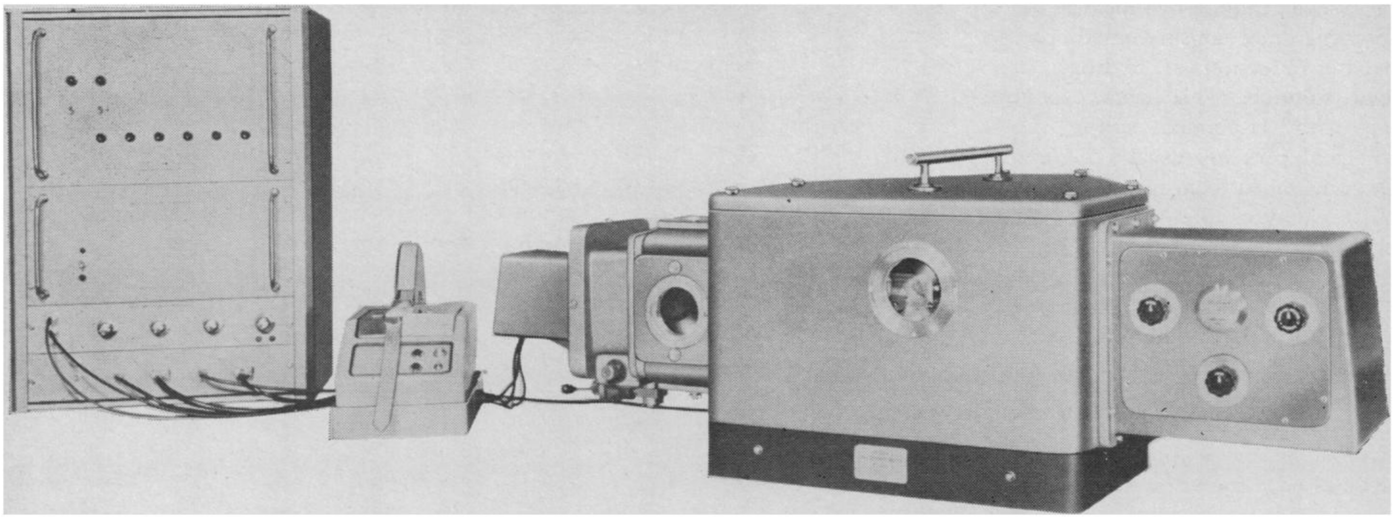
Ruby laser microprobe focuses laser beam on sample through microscope. Vaporization of 50-micron sample is painless, can be used for live specimens.



First Report on Interferometer in Use

The interferometer's capacity for scanning all wavelengths simultaneously is a big advantage in emission spectroscopy but less of an advantage in making absorption spectra, according to Arthur Maki of the National Bureau of Standards. Maki reviewed his experience in using an interferometer over the last year. In looking at absorption spectra, he said, the broad wavelength range covered by the interferometer makes it harder to distinguish each absorbed frequency from its neighbors and from background noise, which is resolved at the same level as the spectrum itself. A practical method of eliminating noise, he said, is to run two spectra and superimpose them; the peaks that drop out are, of course, noise. The biggest disadvantage, Maki found, is the cost of computer time, which he said is $8 per minute at the Bureau of Standards. The higher the resolution the greater the cost; Maki said a detailed CO spectrum would cost $350.

The new instrument is not likely to retire grating dispersion spectrometers. Clive Perry, a young British physicist who has recently become an associate of Richard Lord's at M.I.T. and who is studying the unusual properties of perovskite crystal structure, said he would "probably use the interferome-



British-made interferometer is described as yielding high resolution over wide frequency range in the far-infrared.

ter as a survey instrument, then—once I've found the bands I'm interested in —use the grating spectrometer for further study, then turn back to the high resolution of the interferometer for detailed study of a specific frequency range required. I believe we will get from 3 to 5 times better resolution from the interferometer than from a comparable grating instrument."

Many users believe that far-infrared instrumentation depends in a critical way on the improvements being made in detectors. Charlie E. Jones of Texas Instruments Inc. presented some experimental results indicating that a gallium-doped germanium bolometer operating at liquid-helium temperatures had a signal-to-noise ratio about three times that of a Golay cell over the 100 to 40 cm^{-1} range. The bolometer and helium Dewar are manufactured by Texas Instruments.

Light on the Helix

Like such biological polymers as proteins and nucleic acids, the carbon chains of many man-made polymers curve into a helical structure. The intricate analysis of bond vibrations and rotations in high polymers may eventually throw some light on why the helix seems to be life's essential shape.

While x-ray diffraction studies have disclosed the structures of a few helical molecules of great complexity (for example, DNA, hemoglobin, myoglobin), infrared spectroscopy has established the characteristic frequencies of bond vibrations for certain simpler helical molecules. Spectroscopic analysis has been simplified by the extent to which a helical structure hinders free rotation around the C–C bonds of the polymer's carbon structure. Vibration frequencies have been assigned with assurance to crystalline sections of a number of polymers, but complete crystallinity is not achieved.

In analyzing certain fibrous polymers, Samuel Krimm of the University of Michigan has assigned infrared absorption frequencies to bond structures in noncrystalline sections of the carbon chain as well as in crystalline sections. He told the conference that he had been able to determine, for example, that the stretching frequency of the C–Cl bond in noncrystalline chain sections of polyvinyl chloride is 620 cm^{-1}, while the same bond in crystalline chain sections has a frequency of 610 cm^{-1}. This delicate resolution indicates a differing order of Cl and H atoms along the carbon chain, and the change in configuration alters chain conformation, interrupting the spiral. (Similarly, variation in the sequence of amino acids along a hemoglobin chain may break the helical structure, and 60 to 70 percent is the highest helical content so far obtained.)

In many cases, the higher C–Cl frequency established by Krimm can be used to indicate changes in configuration in unknowns having such bonds, and the knowledge makes it possible to predict how a chain will bend.

"We now have a very detailed handle as to what is going on when you do certain things to a polymer," Krimm said, and he expressed confidence that complete vibration analysis of the noncrystalline as well as the crystalline sections of high polymers would soon be possible.

Scarcely a handful of men all over the world are working in the difficult area of high polymer vibration analysis. Krimm's experiments have extended over 10 years, and he has used the Perkin-Elmer 421 and 301 high-resolution infrared spectrometers.

Polarography and Muscular Dystrophy

Only recently introduced in biochemical analysis, polarography (7) has proved to be a highly specific technique for identifying trace metals of metabolic importance. Because molecules or ions exchange electrons with the mercury microelectrode of this system, it can also be used to follow small changes in oxygen concentration in very small samples and is competing with the Warburg apparatus for research use.

W. C. Purdy of the University of Maryland spoke on study of selenium by this technique. The toxic element in "loco weed," where it is moderately concentrated, selenium has recently been found to be essential as a trace element for utilization of vitamin E in sheep. Without it, sheep get white muscle disease, which resembles muscular dystrophy in humans. Brewer's yeast is effective in reversing white muscle disease; other yeasts are not. Polarography has disclosed that, while selenium is highly concentrated in several yeasts, only in brewer's yeast is it found in the plus-4 oxidation state.

Polarography can also report protein concentration by means of an interesting mechanism based on the catalytic reduction of hydrogen in a cobalt solution. This works for proteins with H-bonded sulfur groups (biological proteins usually contain at least one sulfur-bearing amino acid). Cobalt catalyzes the reduction by replacing hydrogen in the sulfhydryl group, and the reduced hydrogen is detected by electron exchange with the mercury drops of the microelectrode. The presence of sulfur-containing amino acids is apparent in the polarogram as a double wave, Purdy said, and a difference in the nature of the double wave has been found for serums of normal and cancer patients. This difference has been expressed in terms of a "protein index," based on constants of temperature, mercury drop time, and so on. Cancer and certain other diseases raise the index; cirrhosis of the liver gives a lower-than-normal index.

Another application is in study of drugs that may protect against x-radiation. A possible mechanism for drug action might be the removal of trace elements (by complexing), which stops the reactions they catalyze, Purdy said. For example, one of the first body systems to show that an animal has received a lethal dose of radiation is the tyrosine-oxidase system, controlled by a copper-containing coenzyme.

A new square-wave polarographic analyzer being made by Mervyn, a British firm, was shown at the meeting by the U.S. distributor, Matheson Scientific. This instrument reduces the two polarographic components to a compact desk-top unit which sells for $8500 to $9000 and detects trace elements in a ratio of 20,000 : 1.

High-Resolution NMR Studies

When a simplified spectrometer produced by Varian Associates opened nuclear magnetic resonance techniques to organic chemists, many recognized NMR as a shortcut to functional group determination. Based on the discovery that an applied resonance frequency will change the spin orientation of protons in a magnetic field, the technique is now almost as widely used for analysis as infrared spectroscopy.

An advancing NMR field is high-resolution study in which spectrometers differentiating spectral lines less than one milligauss apart are used. Physical chemists are using high-resolution NMR to study fundamental aspects of molecular structure: to trace the means by which a molecule is built up by interaction between nucleus and electrons and by further interaction between nuclei. NMR provides an experimental means of determining whether equations derived from quantum theory hold for such interactions.

One question is that of determining the sign of spin-spin coupling constants (J values); theory predicts an absolute sign, but many experiments have indicated that sign may vary. In a paper given at the conference, Keith A. McLauchlan of the National Physical Laboratory of Great Britain showed how absolute sign of spin coupling for interacting protons may be obtained by comparing it to the sign (calculable) of the direct dipole-dipole interaction between polar molecules oriented by an applied electric field.

Under patent rights assigned by Nobel laureate Felix Bloch and others, Varian Associates is the major producer of nuclear resonance spectrometers in the U.S. A price reduction of almost 50 percent since 1961 has brought the analytic instrument (A60) within the reach of an increasing number of chemists, and a surprising number of orders have recently been received from underdeveloped countries —for example, Kenya. This year Varian introduced the A56-60 spectrometer, which provides a stabilized field for study of fluorine nuclei as well as for hydrogen (proton) studies. Field stability makes it possible to take a rapid and exactly repeatable spectrum on a precalibrated chart, and so increases NMR's analytic usefulness. The A56-60 is priced at $38,500.

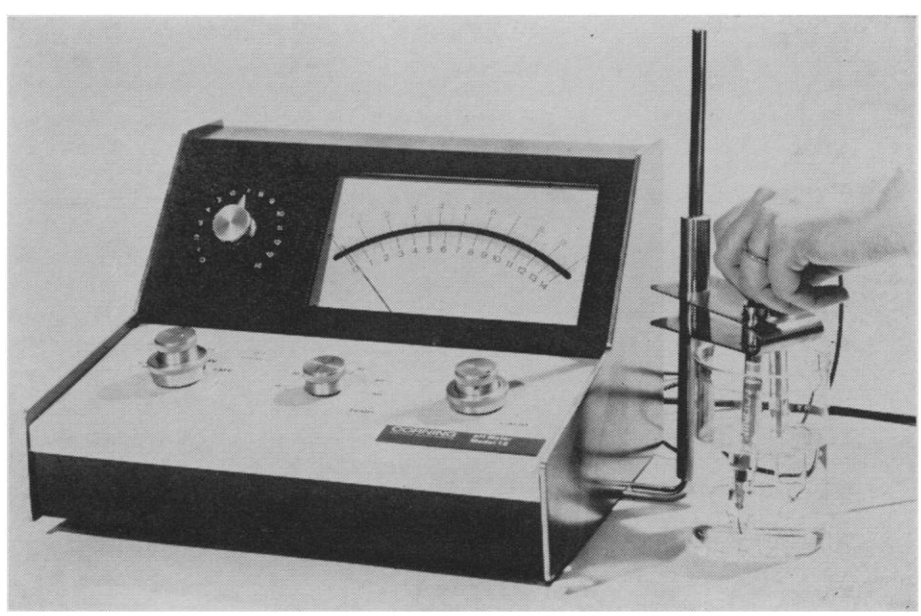
Electron Spin and Photosynthesis

Spinning much faster than the proton, the light-weight electron is also a much stronger magnet. Changes in orientation of spin of unpaired electrons in a magnetic field are produced by resonance frequencies in the microwave range, and spectrometers are available to detect electron absorption of the applied electromagnetic energy. A group headed by Barry Commoner at Washington University, St. Louis, was the first to apply electron spin resonance in the study of the formation of free radicals, or unpaired electrons, in living tissue. Increased sensitivity of ESR instruments has since provided decisive evidence in support of Lenore Michaelis famous conclusion, in 1946, that "all oxidations of organic molecules, although they are bivalent, proceed in two successive univalent steps, the intermediate being a free radical. . . ."

Among other interesting applications are study of photosynthesis, of the effect of radiation on living cells, and of the metabolism of tranquilizing drugs such as chlorpromazine.

A report from Tetano Okabe and Paul Y. Sakagishi of Hitachi, Ltd., in

Corning pH meter uses dual-purpose glass electrode to cover entire pH range.



Japan was read by a representative of Perkin-Elmer, which markets certain Hitachi-made instruments in the U.S. The Japanese researchers said that they had obtained, in an atmosphere of liquid nitrogen, ESR signals from mitochondria and microsomes separated from rabbit-liver cells, and that they were able to differentiate signal values for each type of organelle.

Varian showed a new accessory in use with its ESR 4502 spectrometer. This is a computer which increases the signal-to-noise ratio by adding up a number of scans. The principle is simple. While individual scan signals may be so small as to be difficult to distinguish from noise signals, they are always positive, and therefore additive; noise signals, both negative and positive, will usually average out. Computer averaging of 100 scans increases signal-to-noise ratio by a factor of 10. The computer, which can be used with any field-stabilizer NMR or ESR instrument, is made to Varian specifications by the Mnemtron Division of Technical Measurement Corp. and sells for $11,500.

A newcomer to the instrument show, Japan Electron Optics Laboratory Company, Ltd., showed an ESR spectrometer new to the American market. JEOLCO described this as having a sensitivity of 1×10^{11} spins per gauss at a resolution differentiating spectral lines only 10 milligauss apart (comparable to the resolution claimed for the Varian 4502). The instrument uses an ingenious device to stabilize the external magnetic field, a design solution intended to compete with the Fieldial, patented by Varian. Introduced about a year ago, the Fieldial achieves a stable field by the "Hall" effect: A change in the magnetic field changes the resistance of a single, specially grown crystal, and the resistance change is used as an error signal to correct the field (the patentable contribution of Varian researchers was the growing of a crystal insensitive to temperature change yet sensitive to magnetic lines of flux). The Fieldial makes it possible to dial a magnetic field at a precise intensity anywhere between 0 and 40,000 gauss.

The JEOLCO instrument controls the main magnetic field by controlling the local field of a small reference sample, which is placed some distance away from the experimental region of the field and within an auxiliary set of field-sweeping coils. A microwave frequency is also applied and, at resonance, changes the spin orientation of the reference electrons. The output of a phase-sensitive detector, which is proportional to any shift of the net magnetic field from the point of resonance, is amplified and fed back to the exciting current regulator that controls the main magnetic field. JEOLCO also showed a compact free-radical detector at $32,000.

Among other new instrumental developments reported or shown at the conference were:

► A detector for use in gas chromatography based on measurement of changes in the velocity of ultrasound as it passes through a binary gas mixture. This was developed by F. W. Nobel, K. Abel, and F. W. Cook at the National Institutes of Health.

► A digital data recorder made by Perkin-Elmer for reducing analog data to a form suitable for computer input. The system is designed for use with Perkin-Elmer ultraviolet, visible, and infrared spectrometers, and is adaptable for such spectrometers made by other firms, also for mass spectrometers and gas chromatographs.

► A superconductor magnet developed by Westinghouse with a field intensity of 30,000 gauss and a field uniformity of $1/10^6$. This is a portable unit weighing 90 kilograms (a comparable conventional electromagnet would weigh more than 5½ metric tons), which uses the ductile niobium-zirconium alloy developed by Westinghouse. Westinghouse has recently announced a new superconductive alloy of niobium-titanium which makes cryogenic magnets commercially available up to 80,000 gauss, with 100,000 gauss in sight. Several chemical companies are reported to be working on the possibility of using magnetism to influence chemical reactions.

► A *p*H meter with advanced electrode design, marking the entry of Corning Glass Works, well known for research in electrode glasses, into the analytic instrument field.

T. L. CAMPBELL

American Association for the Advancement of Science, Washington, D.C.

References and Notes

1. I.B.M. provides $108,000 of the monthly commercial lease rate as a grant to M.I.T.; M.I.T. leases additional equipment.
2. Residues taken from suspects' hands or clothing may, after irradiation by neutrons, show radioactive antimony or barium isotopes in the same percentage found in primer powder. The FBI declined to answer a question from *Science* as to whether this analysis had been made in the case of Lee Oswald. Theoretically, such analysis might link a given suspect with a given gun, but for a number of practical reasons such linkage has not been possible except for suicides where scrapings can be taken from both hand and gun immediately.
3. *Rev. Mod. Phys.* **36** (Jan. 1964).
4. E. D. Palik, *Proc. Far Infrared Phys. Symp., Naval Ordnance Lab., Corona, Calif., 1964.*
5. C. H. Perry, *ibid.*
6. *Proc. Intern. Conf. Quantum Electronics, 2nd, 1961.* In Gebbie's original instrument the mirror moved on glass ways; in the R.I.I.C. instrument a piston arrangement is used.
7. For an excellent description of this technique see O. H. Muller, *The Polarographic Method of Analysis* (Chemical Education Publishing Co., New York, 1956).