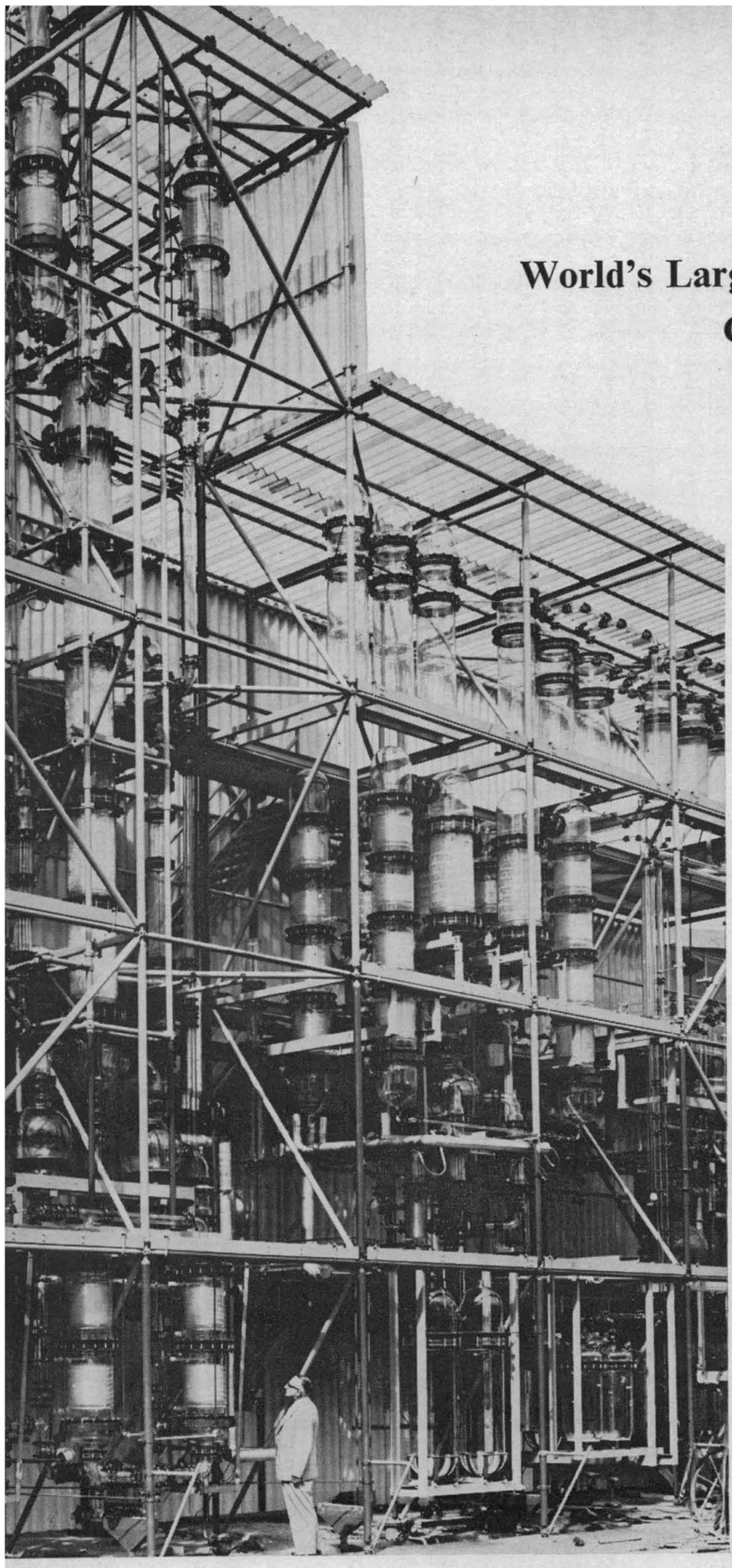


ACHEMA, World's Largest Chemical Engineering Congress, Shows Scientific Instruments of 23 Nations

◄ *Chemical process equipment in glass was erected to a 28-meter height by Jenaer Glaswerk, Mainz. Model of steel chamber for boiling water atomic reactor was set up along main ACHEMA avenue.* ►

The world's largest chemical engineering congress, ACHEMA, arranged by the Deutsche Gesellschaft für Chemisches Apparatewesen, was held in Frankfurt from 18 to 28 June. The site for the congress and the accompanying giant exhibit of laboratory instruments and equipment for the chemical industry was the Frankfurt fair grounds, a 100,000 square meter area where, at other seasons of the year, the German automotive and other industries hold their great trade shows in this commercial capital of West Germany.

The ACHEMA exhibit was a great show of another kind. The many full-sized process installations, and the research instruments designed and manufactured by the 1600 exhibiting firms and organizations, dramatically demonstrated the technological health and creativity that prevail in Europe in 1964. There were exhibitors from 23 nations—from the United States and Soviet bloc countries as well as from western Europe. American firms which have European manufacturing facilities exhibited side by side with trading associations from Czechoslovakia, East Germany, Hungary, Poland, and Yugoslavia in an atmosphere of international

amity. But the countries of western Europe dominated the exhibits. Their displays testified to an impressive industrial and technological resurgence and to the vigorous economic competition that U.S. manufacturers of instruments and equipment now face on the other side of the Atlantic.

Some 130,000 visitors moved along the avenues of the exhibit grounds, through the 18 large exhibition halls, and among the outdoor exhibits. One of the outdoor displays was a high-pressure reactor towering 20 meters and weighing 110 metric tons, built by Badische Anilin & Soda-Fabrik (BASF), one of the three German chemical giants that once composed I. G. Farbenindustrie. Made by winding and shrinking hot steel bands on a cylinder, the reactor was a product of techniques that produce vessels capable of withstanding pressures of 6000 atmospheres.

The famous Jenaer Glasswerk, Mainz, had built an immense process glass installation with modular units—extraction, distillation, separation, and rectification columns. According to Jenaer representatives, the ball flange which permits great flexibility in designing glass installations, including use of glass pipes as large as 60 centimeters in inside diameter, is the property of this company alone. Corrosion-resistant glass equipment, providing complete visibility of operation, is finding ready buyers, especially in developing countries where manufacturers do not have large sums invested in metal equipment.

Also out-of-doors and sparkling in the sunshine was a glass-sheathed heat-exchange unit equipped with 28 tantalum tubes, produced by a French specialist, L'Equipment Industriel en Verres Speciaux, Aubervilliers.

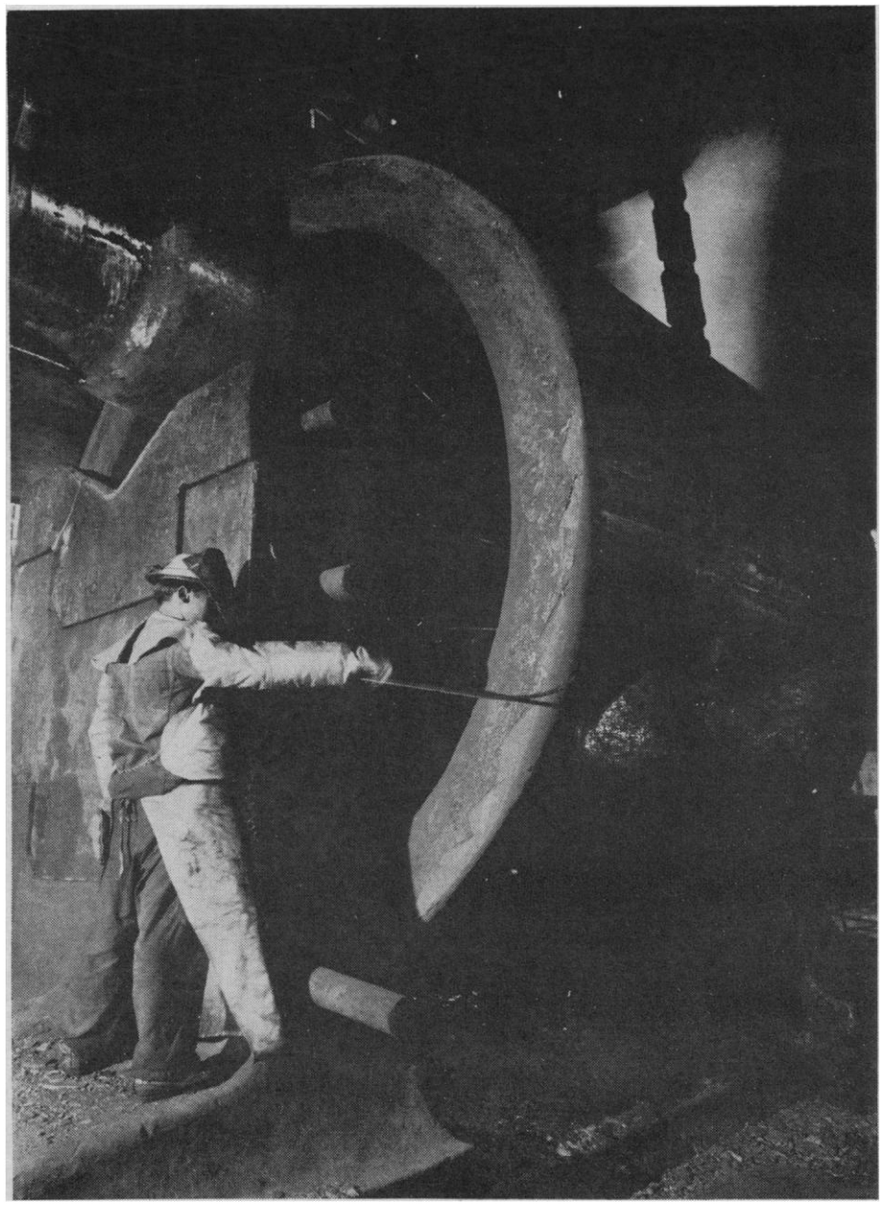
The three large exhibit halls set up by the three companies (BASF, Farbwerke Hoechst, and Farbenfabriken Bayer) that together account for 47 percent of investments in West Germany's chemical industry reflected the prosperity of this sector of the booming West German economy: with $6.5 billion sales in 1963, West Germany ranked fourth among the world's chemical suppliers and showed a growth rate of 7.7 percent.

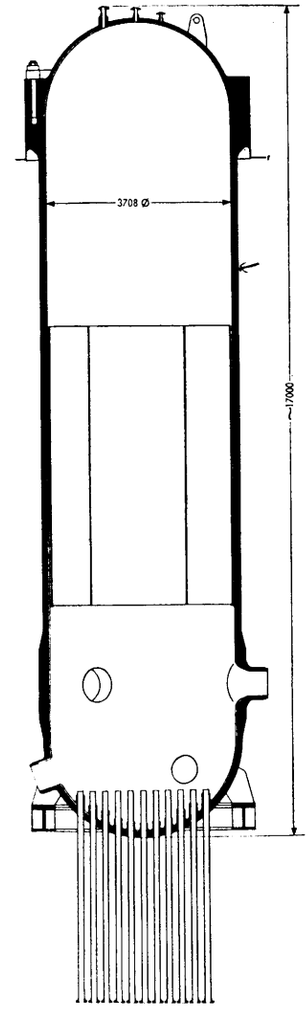
Petrochemical Expansion

The rapid expansion of petroleum-based chemistry in recent years was also evident in these halls. BASF showed a model of a plant it is building at Ludwigshafen which will produce 54,000 metric tons of acetylene a year from hydrocarbons, based on recent development of the BASF partial combustion process to a level that permits use of all light hydrocarbon fractions, including naphtha.

One of the largest plastic parts so far produced was shown by GASF: a Vinoflex (rigid polyvinyl chloride) vacuum filter screen 300 centimeters in diameter, made by welding smaller components together. A recently developed molding technique, BASF representatives said, has made possible economic manufacture of polyethylene filter press plates (1 by 1 meter, in a single piece) and frames. Some 32,000 of these plates, with an operating life expectancy of 2 years, have been installed in a single BASF plant, where they will replace wood plates that have to be renewed every 6 months.

ACHEMA 1964 is the 14th chemical engineering exhibition-congress arranged by DECHEMA (Deutsche Gesellschaft für Chemisches Apparatewesen), which has some 900 corporate members and about 2200 individual members. Karl Winnacker, chairman of DECHEMA's board of directors, is





Vessel for boiling water atomic reactor was forged as a single steel cylinder by Rheinstahl Hüttenwerke. It will be installed at Gundremmigen on the Danube where a nuclear power plant, being built under Euratom program financed by six countries, will produce electricity for international industrial area.

also chairman of the board of Farbwerke Hoechst, the great company founded in 1863 when the chemist Eugen Lucius and his brother-in-law Wilhelm Meister, a Hamburg banker, decided to take a hand in transferring the coal-tar dye industry from Perkin's mauvine pots near Harrow to Germany.

While the task of organizing ACHEMA every 3 years is a major function of DECHEMA, it is not the only one. A new building to house the society's vigorous research program will soon be completed. Some 4000 pages of specifications for materials and process equipment strong enough to withstand the high stresses of modern chemical processing are currently being published, Dieter Behrens, DECHEMA director, said. Much attention is given to education: doctoral candidates are given support to attend DECHEMA courses in process techniques and materials and in instrumentation. ACHEMA has a role in education: exhibitors could be seen giving special attention to the thousands of students who attended in teacher-guided groups.

The European Federation of Chemical Engineering held its 52nd meeting during the congress. The Federation unites 40 scientific and engineering associations from 18 countries and has corresponding members in an additional nine; it has solved nationalistic problems by not having a president.

The European Federation for Corrosion arranged a symposium on new materials for extreme conditions (see page 1035). The Deutsches Atomforum prepared a program on nuclear radiation in science and engineering. American participation in the program was mainly in the sessions on new physical methods of chemical analysis (page 1033) and use of the computer in chemical technology (page 1033). A large audience turned out for a plenary lecture given by Karl Kammermeyer, University of Iowa, who reviewed U.S. progress in space technology with particular reference to chemical engineering achievements.

If ACHEMA gives dramatic evidence of the technological wealth of most of the Western nations, it is also a marketplace where individual fortunes can be made. This is where managers in a highly sophisticated industry, one in which brains are still probably the most significant part of invested capital, get a chance to size up each other's bids for the market in a dozen countries. The scientific instrument industry probably moves faster than any other sector of modern technology; new instruments with enormous commercial possibilities rapidly and unpredictably appear as individual investigators discover new ways to approach the fundamental energies that constitute life and its physical environment.

Compared with the enormous capital invested in process equipment in chemical and other science-based industries, capital invested in research and laboratory instrumentation is small. There is, therefore, every reason for the market to keep pace with the rapid advances in instrumentation, and the manufacturers who survive and grow in this restless field are those who are willing to put their electronic engineers to work at the first hint of a new way of seeing something; such a hint may be given by an obscure paper appearing in one of the world's many scientific journals or presented at one of the thousands of scientific meetings held annually throughout the world.

Thus the instruments to be seen at ACHEMA ranged from devices like the new electron-probe x-ray microanalyzer, which will sell in Germany for $75,000, into which Cambridge Instruments, London, had put years of developmental work and a capital investment estimated at more than $500,000, to a $500 miniature nuclear magnetic resonance spectrometer designed by a young English physicist, Paul Cook, whose initial financial investment was £200.

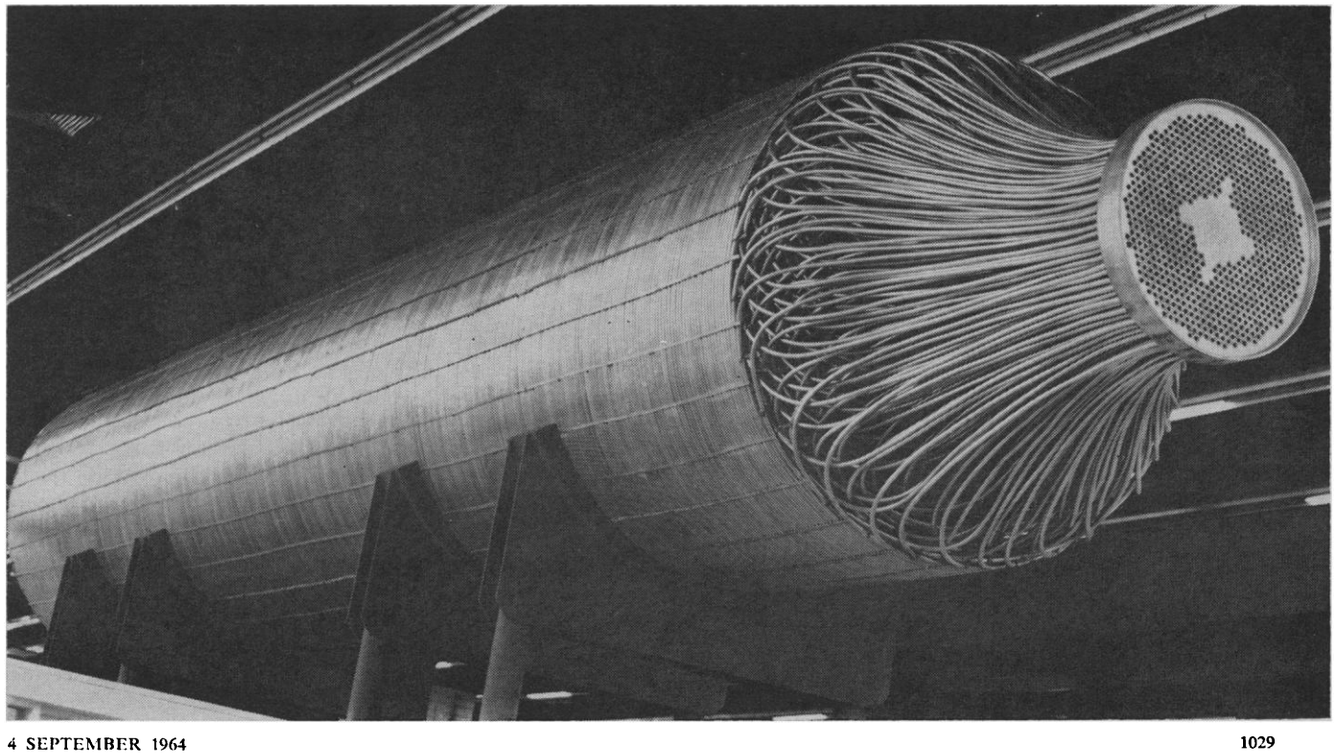
Cambridge Instruments' new scanning x-ray microanalyzer is designed for geological and mineralogical analysis. Developed in collaboration with J. V. P. Long of Cambridge University, and based on his original design, it represents an extension of the technique of x-ray microanalysis, a means of investigating chemical microstructure by spectroscopic analysis of x-rays emitted from the surface of specimens upon excitation by a primary beam of electrons, which has hitherto been largely limited to metallurgical applications. While designed to accommodate large specimens of clay, ceramics, or ore, the new instrument can also be used for studying metals. Cambridge Instruments estimates that it will be able to produce 24 such instruments a year.

Cook's inexpensive instruments, which represent an investment of little more than his own imagination, stole the show at the exhibit of Shandon Scientific Company, Ltd., where he occupied a corner of the space reserved by this large British instrument maker, whose annual sales amount to about $3 million.

Cook's basic idea was simply to reduce the size of the coil in the head of the nuclear magnetic resonance probe, and he accomplished this so successfully that his instrument can detect the fine-structure (wiggle effect) in proton signals with a 7½-centimeter magnet over a range of 400 to 10,000 gauss. High sensitivity at reduced coil size is achieved by immersing the detector coil in the nuclear resonant material, so that the volume normally occupied by the glass sample tube is occupied by the specimen itself.

Cook says he got the confidence to make a start in instrument develop-

Heat exchange is composed of 2785 miles of aluminum pipe. It was made by Gesellschaft Linde, Hattingen (Ruhr) and will be installed in chemical plant for the separation of air into liquid oxygen and nitrogen, to be built in England by Linde's plant construction division.



ment after a visit to the U.S., where manufacturers showed an interest in new ideas. His company, Scientifica, is also producing an electron spin resonance spectrometer in which a low-frequency magnetic field is used with high-frequency modulation to obtain free-radical signals in the presence of water. He intended these instruments for teaching and demonstration at the graduate level, but their sensitivity has already won them a place in research (at Ford Motor Company and London University, England, for example).

Cook is further developing his nuclear magnetic resonance spectrometer, which he believes may have wide industrial application, including purity analysis in the food industry. His most recent design—a recording bacterial-colony counter—is already in use in England's biggest meat plant, and he anticipates that 1964 sales of all his instruments will amount to about £30,000. Shandon is selling Scientifica instruments in Europe; Barnes Engineering, in the United States.

Polymer Dielectric Response

A simple and inexpensive method for detecting impurities in polymers in concentrations as small as 0.01 percent was described in a paper presented at the congress by Peter Hedvig of Plastics Research Institute, Budapest. Use is made of the fact that impurities change the dielectric relaxation time of polymers. The technique is a simplification of the method introduced by Hamon, who recognized that polymer structure and other properties could be investigated by the response of polymeric dielectrics to the application of a square voltage pulse.

This is basically a simple matter: the current response of the dielectric sample to an electric pulse is measured by an electronic microammeter. But to estimate dielectric relaxation time it has been necessary to transform current-response readings into a frequency scale by means of a Fourier transformation method developed by Hamon and Williams and then to read maximum dielectric loss in relation to frequency.

Hedvig's contribution is to show how the change in low-frequency relaxation time can be computed directly from the experimental charge and discharge curves without use of the cumbersome Fourier transformation. Both charge and discharge curves have two components: (i) a sudden change in strength of the electric field, and (ii) a slower change resulting from the low-frequency relaxation time of the dielectric material.

"If the time constant of the electronic microammeter is properly adjusted," Hedvig said, "the amplitude of the curve representing the material's response to the electric current is proportional to the inverse of the low-frequency relaxation time. . . . The response curve is sensitive to both polar (water, alcohols) and nonpolar impurities."

While the mechanism of slow dielectric polarization in polymers is not well understood, Hedvig said the effects observed by this method are probably due to Maxwell-Wagner type dipoles formed at the boundary between impurity and polymer. The method "cannot, of course, compete with nuclear magnetic resonance, for example, but owing to its simplicity and low cost can be very useful for rapid checks on impurities and in studying plasticizing problems."

Cathode-Ray, Twin-Cell Polarograph

A twin-cell cathode-ray polarograph which gives new precision and speed in analysis has been developed by researchers at the United Kingdom Atomic Energy Authority (UKAEA). A similar instrument, developed by Southern Analytical, Ltd., Camberley, Surrey, England, was exhibited by the company and described in a paper given by its research director, H. M. Davis, who was formerly a member of the research staff of UKAEA.

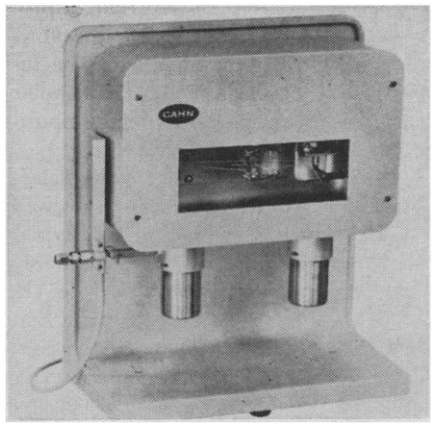
"This method of measurement has the unique advantage that the 'drop wave', which was a major limitation of classical polarography, is completely eliminated," Davis said. "The trace is reproduced exactly from drop to drop and a complete polarogram over the range of 0.5 volts is seen every 7 seconds."

The new twin-cell polarograph also permits what Davis called "comparative polarography," a technique developed by UKAEA researchers. This is based on direct measurement of the difference in polarographic current between a cell containing the unknown solution and a cell containing a reference sample.

"The resultant polarographic wave form may be greatly amplified so that currents corresponding to very small concentration differences between unknown and standard can be measured with high accuracy. From a separate polarogram of the wave resulting from the standard it is then simple to calculate the composition of the unknown."

Davis said that this method had been used by Shalgosky and Watling to de-

Scientific instrument displays filled four floors in ACHEMA's largest exhibit building.



Gravimetric detector for gas chromatography was shown by Cahn Instruments, Paramount, California.

termine U^{4+} and U^{6+} ratios in uranium oxide. Describing successful examination of a number of copper alloys he had made by this means, Davis also said the results agreed well with those obtained in a test with conventional analytic methods. "The chemical determination for copper and zinc required 4 hours per sample as compared with 30 minutes for the polarographic technique."

The advantages of the twin-cell cathode-ray system of polarography were described in a paper given by R. C. Rooney, also of Southern Analytical, Ltd. In the single-sweep instrument, an increasing direct-current potential is applied across the cell in the usual manner, except that it is applied once in the life of every mercury drop.

"Use of a cathode ray tube as the indicator makes it possible to follow rapid current changes easily," Rooney said, "while the absence of drop growth oscillations allows the use of considerably greater electronic amplification than can generally be used, giving increased sensitivity."

Rooney added that the method guarantees a tenfold increase in sensitivity even in the worst cases, while a 100-fold increase is obtained in some reactions.

Cathode-ray polarography makes it possible to use smaller samples than those required in conventional polarography and to omit the chemical separations required by the conventional technique. The former advantage has made the instrument particularly useful in examining radioactive materials, Rooney said. Polarography is being increasingly used in Britain, he said, for examining toxic metals in goods (partly as a result of recent legislation tightening standards) and for determining impurities in materials used in the manufacture of transistors.

Instrument for Atomic Absorption Studies

A spectrometer providing for both flame emission and atomic absorption analysis at the turn of a switch was exhibited by Unicam Instruments, Ltd., Cambridge, England, and was described by Unicam's chief chemist, W. J. Price, in a paper presented at the congress.

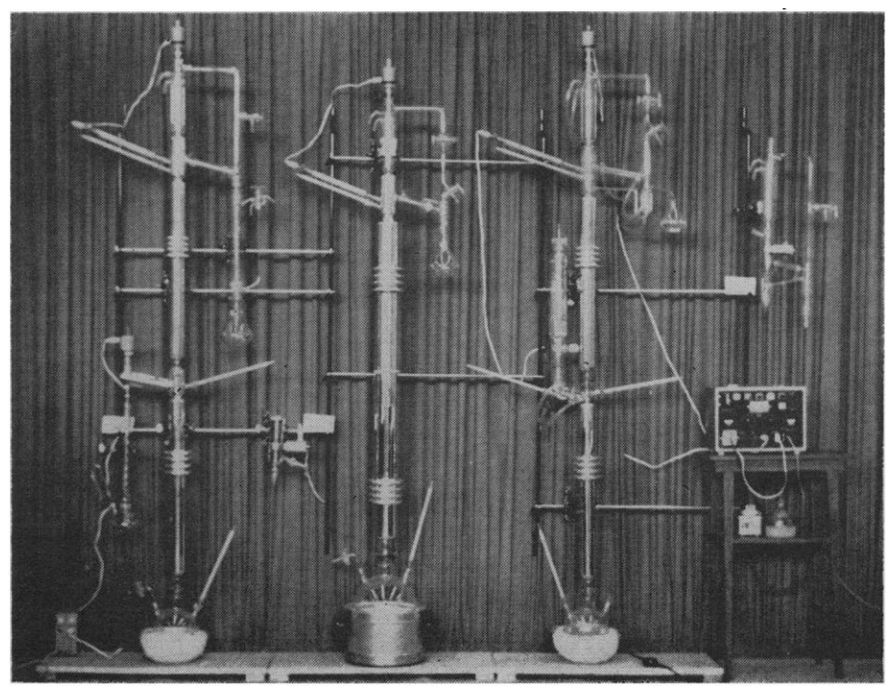
In Unicam's instrument the sample solution is sprayed into a 7-centimeter flame (burners are provided for both acetylene and propane, which provide the lower flame temperature required for absorption analysis of many elements). Some of the atoms in the sample are excited and determinable by the usual emission technique, but many of them remain in the ground state upon disassociation of their compounds. When a beam of monochromatic light of the resonance frequency of the atoms of the element in question is passed through the sample, the proportion of the light absorbed depends on the number of atoms present—that is, the proportion of light absorbed indicates the concentration of the element in the sample.

The light source in the Unicam instrument is a hollow cathode lamp whose cathode incorporates the element under observation. The lamps are designed to plug in as self-contained units and are available for some 24 elements. Additions to the list are being made constantly, Price said, and this leads to an increase in the number of applications.

"This instrument has already been used by biochemical research workers for the determination of minute amounts of Mg and Zn in biochemical fluids," Price said. "In both cases the presence of other substances in great excess precluded determination by other means. Mg contents as low as 0.012 parts per million were measured in the presence of relatively large concentrations of KCl and of PO_4."

While analysis of magnesium in blood serum involves higher concentrations (a pathological level is about 50 parts per million), "classical and colorimetric methods are considered to be impossibly lengthy or unreliable for clinical use," Price said. "Flame emission methods are better, but require the addition of fairly complex radiation buffers in order to minimize the

Automated distillation unit was shown by Normag of Hofheim, Germany. Microcell Filter Co., Grosse Pointe Farms, Michigan, immediately made arrangements to distribute it in the United States.



interference effects of other serum constituents. Such interference does not occur in the absorption method. Further, the sensitivity is so much higher that greater dilutions of the sample may be used and this both 'dilutes out' cationic interference effects and minimizes difficulties in atomization. Depressive effects of protein and PO_4 anions are completely removed by the addition of strontium as a releasing agent."

Price also mentioned the value of the instrument in metallurgical determination of magnesium concentrations. The fact that graphite can be induced to form as spheroids in cast iron in the presence of 0.01 to 0.1 percent of magnesium under certain conditions makes the analysis important. "Assuming a sample dilution of 1:100, this reduces to an analytical problem of determining Mg in the range 1–10 ppm in the presence of a 1000- or 10,000-fold excess of iron. Chemical methods require removal of most or all of the iron by electrolysis or solvent extraction, and the magnesium concentration is determined either by precipitation as a phosphate or by titration. The atomic absorption determination of Mg is quite specific even in the presence of such large excesses of iron—in fact, iron appears to lower PO_4 interference."

Zeiss, Leitz, and Others

Germany's great optical firms were well represented. Among a great variety of instruments, Carl Zeiss (Oberkochen, Germany) showed a prototype spectropolarimeter which it expects to have on the market in about a year. The optical activity which steroids and other organic compounds show in the ultraviolet range has been increasingly used as a key to structure since O. C. Rudolph & Sons introduced the first commercial spectropolarimeter, a manually operated instrument. Applied Physics Corporation (Monrovia, California) for about 2 years has offered a precise recording instrument designed by Howard Cary; so far it has installed a number of these at a cost of about $40,000 each.

The Zeiss instrument has a wavelength range similar to the Cary instrument, 180 to 600 millimicrons. Both instruments were operating briskly at ACHEMA, and the two manufacturers had both chosen the sharp optical rotatory dispersion curve of camphor for the demonstration.

Zeiss also showed a new color-measuring instrument which combines a monochromator with a photometer and provides shadow-free illumination of the sample. Color values are automatically computed in terms of the three basic color components of the spectrum. Hans Prugger, a physicist and former Fulbright scholar at the University of California, who is now associated with Zeiss, said the new instrument provides an absolute color measure and differs in versatility from the color-measuring instrument built by Hardy in the United States. Zeiss sold its first color register, at about $30,000, to the Federal Institute for Material Testing in Berlin for research in color pigments and for color standardization work.

Ernst Leitz (Wetzlar, Germany) showed its microspectrograph equipped for direct TV observation. Described by the manufacturer as having optics of "almost ideal achromatism," making the optical conditions virtually independent of the wavelength being observed, the instrument permits measurement in the ultraviolet as well as the visible region of both absorption spectra and of fluorescent emissions from specimens less than 1 micron in size.

A built-in phase-contrast system, with its own eyepiece, is added for focusing and adjusting objects in the slit of the microspectrograph. The image orthicon tube of a television camera can be used in place of the spectrographic plate, permitting representation of both object and reference spectra and of monochromatic object image on the screen of a television receiver as amplified video signals. A line-selector-switched oscilloscope presents the signal of any line of the television image on the oscilloscope screen. Television observation is particularly valuable for spectral measurement of rapid events, such as occur in living systems. Very weak signals can be greatly increased by extending the storing time between two scanning processes in the image orthicon tube.

Andreas Thaer, a member of the Leitz research staff, said he expected the instrument to enable researchers to follow some of the chemical reactions in living cells. The equipment could also be used for damaging a minute part of a living cell—for example, a part of the nucleus by ultraviolet radiation—and then observing the effect on the functioning of the cell.

The Leitz microspectrograph was designed in collaboration with F. Ruch of the Federal College of Advanced Technology, Zurich. The combination with highly sensitive TV equipment follows a system described by S. S. West of Western Reserve University, Cleveland, Ohio.

Dow Chemical is the first U.S. customer for a double-focusing mass spectrometer exhibited by Atlas Mess- und Analysentechnik, Bremen. According to the manufacturer, this instrument has a sample inlet system superior to that of most instruments now on the market. Because of its high sensitivity, only minute samples are required. In this instrument interchangeable cells provide for introduction of liquid, gas, or solid samples in an insulated chamber that maintains the sample at a constant temperature independent of the temperature of the ion beam through which it is passed. This feature is particularly important in studying organic samples that decompose easily.

Researchers at Britain's Institute of Cancer Research (ICR) are using a new steady-state distribution machine to separate RNA fractions. This instrument was shown by Quickfit & Quartz, Ltd. (Stone, Staffordshire, England), a great glassmaker which rivals Jenaer Glass in the production of large process installations.

According to the ICR research, it takes some 2433 transfers in this instrument to separate components of a 100-milligram sample of RNA fractions to 99-percent purity. The machine can also be used for concentration and continuous preparation of compounds.

The instrument is described by its manufacturer as an improvement over the countercurrent extraction method devised by Lyman Craig of the Rockefeller Institute, in which components of a mixture distribute themselves according to their affinity for different solvents. The Quickfit machine, based on a system of steady-state distribution with discrete stages first described by Alderweireldt (University of Ghent, Belgium), is said to be faster and more flexible than machines based on the Craig method, which involves a search for the best solvent system.

The automatic performance of the Quickfit instrument is directed by a controller programmed for a desired ratio of upper-phase to lower-phase transfers. A simplified mixture discharged from the long train of glass cells after initial separation may be reintroduced and further separated, with the same solvent system but a different program.

An automated oxygen analyzer, shown by Coleman Instruments, Maywood, Illinois, attracted considerable interest among analytical chemists because it replaces a cumbersome analytic train which usually spreads over an entire laboratory and requires 3 to 4 days for consistent results. While the automated process is still microchemistry, determinations require 4 hours or less. The combustion analyzer automates the Unterzaucher method of oxygen determination by means of a cyclical control system.

Coleman also showed for the first time a macromolecular nitrogen analyzer, useful for analyzing nonhomogenous samples of up to 500 milligrams, such as animal feed and fertilizers. Like the micromolecular nitrogen analyzer (used for samples of 5 mg or more) introduced by Coleman in 1960, this instrument is based on automation of the classical Dumas combustion method. The manufacturer says that either instrument permits four to five determinations per hour, as compared with the slower Dumas or Kjeldahl methods.

American Plants Abroad

American scientific instrument companies that have established European manufacturing facilities were prominent in both the program and the exhibit area, while the total number of U.S. companies participating was larger than in any previous year.

Many of the Americans who presented papers dealt with gas chromatography. L. S. Ettre of Perkin-Elmer, Norwalk, Connecticut, reviewed new developments in column technology, including two-column processes, neutral carrier substances for analyzing polar samples, and variation of carrier material to eliminate secondary absorption effects. Perkin-Elmer has a large manufacturing plant near Lake Constance, and this plant, Bodenseewerk Perkin-Elmer, arranged a large exhibit at which an automatic polarimeter equipped with a gas laser was a center of interest.

Beckman Instruments, which has a manufacturing plant at Munich, also had a large exhibit, showing a full line of spectrometers and chromatographs. F. & M. Scientific Europa, which manufactures in the Netherlands, showed gas chromatographs as well as its well known carbon-hydrogen-nitrogen microanalyzers. A fourth U.S. company, Wilkens Instrument (Walnut Creek, California), recently established an assembly plant near Amsterdam and expects to set up manufacturing there.

Companies in England are now the largest suppliers of scientific instruments to Communist countries. (Bodenseewerke Perkin-Elmer, for example, is forbidden by the terms of the contract under which it makes special equipment for NATO to sell to Soviet-bloc countries, but the Perkin-Elmer plant in England is free to do so.) Thus, British companies were particularly interested in sizing up the instruments exhibited from eastern European countries.

With the exception of some former Carl Zeiss facilities in East Germany, the Communist countries represented at ACHEMA, it is believed, do not yet have the optical basis for manufacturing spectrometers comparable to Western instruments. But Kovo (Prague) showed a recording gas chromatograph equipped with flame ionization, thermal conduction, and argon detectors and described by specifications that suggest real competition for similar British-made instruments in the home market. There was no indication, however, that Kovo could sell at a price that would be competitive in western Europe.

New Trends in Gas Chromatography

A new dimension has been added to gas chromatography by simultaneous use of flame and electron capture detectors at one column outlet, according to a paper given by K. P. Dimick, of Wilkens Instruments. The effluent gas is divided by a splitter, and the two streams feed the two detectors. The flame detector responds to almost all volatile organic compounds, while the electron-capture detector is sensitive only to electronegative compounds. The signal from each detector is separately amplified to drive a two-pen recorder system.

The ratio of the two signals is unique for a specific compound and thus serves as a means of identification. The system also identifies anomalous results, often obtained by use of the electron-capture detector alone. Dimick said that Wilkens researchers have successfully used this technique in identifying components of a number of natural products; signal ratios found have varied over a range of 100,000.

A relatively new technique, the electron-capture detector, uses a tritium source of about 250 millicuries. "Unlike other detectors," Dimick said, "it measures the loss of signal rather than a positively produced electrical current. As the nitrogen carrier gas flows through the detector, the tritium source ionizes the nitrogen molecules and slow electrons are formed. These slow electrons migrate to the anode under a fixed voltage. Collected, they produce a steady current to the electrometer. If a sample containing electron absorbing molecules is then introduced, this current will be reduced. The loss of current is a measure of the amount and electron affinity of the compound."

The two-channel (two-detector) technique "portends tremendous usefulness in both the biochemical and biomedical fields," Dimick said.

A chromatographic technique in which columns packed with specially prepared polystyrene gels are employed for determining the molecular weight of high polymers was described by Lawrence E. Maley of Waters Associates, Inc., Framingham, Massachusetts. Polymer fractions of very similar molecular weight have been successfully separated by elution with aromatic and chlorinated solvents through the gel-packed columns in 1 to 3 hours. This is a tedious undertaking by the usual column fractionation techniques. In the gel-column system, effluent concentrations are detected and recorded by a continuous differential refractometer.

Computer Control of Chemical Processes

The major U.S. contribution to the program was made at the sessions on the use of the computer in chemical technology, where six Americans gave papers.

Since the Texas Company installed, in 1959, the first digital computer to be assigned to a process control task in the worldwide chemical and petroleum industry, some 120 digital computers have been installed for this purpose throughout the world. Theodore J. Williams, Monsanto Company, St. Louis, said that most of these are supervisory computers connected to conventional plant control systems. Now a new class of control computer is beginning to appear "economical enough, flexible enough, and of sufficient reliability to be used as actual in-line plant control elements."

The first direct digital control (control in which the computer is used to move the process valves directly without going through a conventional control system) of a full-size chemical plant was installed in 1962 by Imperial Chemical Industries in a soda-ash plant in Lancashire, England, using the Ferranti Argus 200 computer. Encour-

aged by this news. Monsanto, DuPont, and the 3-C project (Case Institute of Technology, Conoflow Corporation, and Corning Glass Works) stepped up their tests of direct digital computer control. These studies led to a set of specifications which were revised at a workshop made up of delegates from 20 potential user companies and issued to all firms interested.

Both Monsanto and the Esso Research and Engineering Company have announced that they will make full-scale plant installations of direct digital control in the near future, Williams said. Such computers "open whole new areas of plant control schemes for exploitation since the computer allows an almost complete freedom of choice of control methods for coordination of plant variables and for optimization of plant operation."

Direct digital control systems are being designed with and without the capacity for what designers have termed "optimization." This means that the computer will have the capacity to determine a new optimal operating level in response to changing conditions. Williams said such facilities are of two types:

1) Steady-state optimization, which assumes that the process is at a steady state and can be instantaneously transferred from one steady-state level to another—an assumption necessary for transforming process operation equations to the algebraic form needed for solution by most existing computer systems.

2) Dynamic optimization and control, which adds one more level of sophistication.

Dynamic optimization programs are based on mathematical models composed of sets of simultaneous differential equations, in contrast to the algebraic equations of the steady-state optimization model. Williams described such programs as "mainly of academic interest at present due to the extremely large and powerful computing capacity required to achieve them" but said that "practical attainment in the foreseeable future is highly possible." Such programs, he said, would find particular application in cyclical catalytic processes and in the batch processing of plastics.

Direct digital computer control (which can eventually put all the central control equipment for a medium-sized petrochemical plant in a space equivalent to about three file cabinets and a large desk) promises, according to Williams: (i) Cost of about two-thirds that of a conventional electronic or pneumatic control system, for a large chemical or petrochemical plant. (ii) Reduction of total personnel to a fraction well below present needs. (iii) Elimination of much of today's plant accounting department. (iv) Dependability; at most the time during which the automatic control system would be inoperable because of system failure would be less than 4 hours per year.

Realization of these potential advantages, Williams said, waits only upon the completion of the new control instruments by several manufacturers now at work on the basis of specifications provided by the chemical companies, and upon the "development of standard programs for logging, alarming and control to avoid most of the high engineering costs that would be involved if each customer did all his own programming."

Hybrid Computers

While the digital computer uses discrete numbers and achieves great speed in operating with these integers on a sequential basis, the analog computer simulates a specific process and considers all operating variables simultaneously. Thus, the analog computer gives a continuous output, which may be read constantly, to show the result of the several variables, as a mercury thermometer shows the temperature. Analog computer control of chemical processes was discussed in a paper prepared by Donald F. Othmer (Polytechnic Institute of Brooklyn, New York) and Takeshi Utsumi (Mobil Chemical Company, New York) and read by Othmer at a plenary session.

The resistances, inductances, and capacitances of electrical circuits, coupled with amplifiers, are used to simulate process variables. Deviations from optimum conditions are immediately indicated by the analog, and the error signals are transmitted to the final control elements, such as valves. The analog has advantages as a controller in processes requiring a throttling action, Othmer said, but it "is somewhat more difficult for the analog to make final decisions based on indicated variations from a desired pattern because of its bulky relay elements compared to the tiny memory core in the digital computer."

Many special-purpose analog computers are being used successfully for control of single process steps in chemical plants. They are practical even for the simplest one-column continuous distillation operation, which requires four mechanical controls to operate at maximum capacity and minimum cost, Othmer said.

Although it is less precise than the digital controller, the analog costs much less and is "particularly good where continuous indications and controls are desirable and where the mathematical model is incompletely understood, or may have differentials of different orders, and/or integrals."

The advantages of each type are being combined in "hybrid" computers, which are "especially valuable for the optimization of profit functions with many parameters and many independent process variables," Othmer said.

Telefunken, Ulm, described by K. H. Adler of its research staff as the only company in Germany to be working on hybrid computers, expects to deliver its first hybrid unit to the Technische Hochschule in Stuttgart for research use by the end of this year. The hybrid will be a composite of Telefunken's big digital TR4 (slightly smaller than the IBM 790) and the analog unit, RA 800. Telefunken recently installed an analog computer for Bayer in a plant near Cologne; it will be used to investigate reaction kinetics and other problems.

Analog computers have been used with special success as guides to the design of exothermic reactor systems, in which temperature rises may cause rapid catalyst deterioration, damage to the reactor, and loss of yield, according to E. B. Kretzmer of Esso Research & Engineering Company, Florham Park, New Jersey.

Describing a simulation study of this sort, Kretzmer showed how the mathematical model described the process by equations representing material and heat balance, basic types of control, and alternative system arrangements. When these were programmed on an analog computer, the designer had, in effect, "an operating reactor at his fingertips that would perform just like the real thing."

High Payout Rate

A paper prepared by G. L. Farrar, engineering editor of the *Oil and Gas Journal*, Tulsa, Oklahoma, reported on the journal's recent survey of the use of computer controls in petrochemicals and refining. (The paper was read by J. W. J. Koenig of Esso AG, Hamburg.)

Some good work has been done in dynamic control, Farrar said. "An IBM–Du Pont joint study team has achieved dynamic control of a cyclic, nonlinear acrylonitrile process ensuring an optimum path from startup to shutdown."

Payouts on computer installations, ranging from less than 1 year to 3 years, are exceeding expectations when indirect benefits are realistically assessed, Farrar said. "One company showed $465,000 per year direct saving, but an actual payout of $1 million annually, when indirect benefits were included."

The Journal's survey found 27 announced applications of digital computer control in refineries (computers installed, or installations definitely planned; since this industry is notoriously close-mouthed, actual applications doubtless exceed this figure). American Oil has five control computers, Phillips Petroleum has four, Standard Oil of California has three.

Materials for Extreme Stress

New materials developed for spaceflight and nuclear technology are being increasingly used to withstand high temperatures and pressures in chemical process construction, I. L. Hepner, editor of *Chemical and Process Engineering*, London, said in a paper on this subject.

Nickel alloys developed in the design of gas turbines are also being widely used. The method is to replace iron, in an iron-base alloy, with nickel, and, for operating temperatures above 1000°C, to replace nickel with cobalt; chromium is added to prevent oxidation.

Where the physical properties of nickel are desired but its mechanical properties are a handicap, a nickel matrix in which submicroscopic thoria particles are dispersed is being used. Dispersion hardening, a versatile process developed by DuPont, can also be applied to copper, iron, and aluminum, Hepner said.

A development recently announced by G. J. Morley of the Rolls Royce Company will probably have revolutionary repercussions, according to Hepner. This is a composite consisting of fused silicon fibers embedded in a matrix of aluminum. The strength of the fibers almost approaches the theoretical limits. An applied load to this matrix will be carried by tensile strength in the fibers and transmitted by shear stress in the matrix. Hence the composite matrix is extremely strong over a very wide range of temperatures.

Pyrometallitimides have been developed which show only small degradation up to 600°C, and Hepner said that some researchers believe many other aromatic cyclic polymers will be found to have similar resistance to high temperatures.

Cermets (composites of ceramics and metals) are prepared by means of solid-state reactions at a temperature range of 1000° to 2000°C and fabricated by hot processing (sintering). These tough, difficult-to-machine materials, hitherto used chiefly in the rocket-propulsion and nuclear industries, are now being used in high-temperature process chemistry in the manufacture of seals, valve seats, and other parts.

Matco, Ltd., has developed a new cermet for coating parts of jet engines and rocket exhausts, Hepner said, which has the temperature resistance of base metals, the bonding characteristics of nickel alumide, and the refractory characteristics of ceramic materials. Recrystallized graphite used in rocket nozzles and resisting temperatures up to 3000°C also has a considerable future in the chemical industry.

Protecting Air and Water

A major part of the ACHEMA program was devoted to consideration of the chemical industry's responsibility for dealing with air and water pollutants. In a plenary-session lecture, O. Jaag, director of the Institute of Water Purification, Zurich, called these "a threat to the health and life of the whole population."

"The lakes and rivers in the industrial nations of the world are veritable open sewers," M. R. Domras, Schwetzingen, said at this session.

The chemical industry is using two main approaches in treating polluted water, Domras said: (i) purification and reuse, particularly significant in view of dwindling water resources, and (ii) introduction of biological systems for metabolic breakdown of complex organic pollutants. Domras described a number of industrial installations and referred to the Merck & Company system at Elkton, Virginia, as a "sophisticated biological treatment plant."

Jaag said that the "metal industries in the Federal Republic of Germany have seen of late the advantages of such recirculation of water which entails saving in clean water and a quantitative reduction in sewage." Wastes from chemical pulp plants and paper mills are also now being reclaimed "to the advantage of surface and ground water protection."

The great European rivers flow across many national boundaries, and thus the problem of water protection is more difficult in Europe than in the United States. For 14 years the International Commission for the Protection of the Rhine, whose members are five nations traversed by the river, have sought a solution, but cost has precluded the adoption of technically feasible proposals, such as pipelines to carry wastes to the sea or rubble storage of waste salts.

Pollution of the Rhine begins in the Lake of Constance region; while downstream French potassium mines and soda plants pour out waste salts. Pollution continues through the industrial sectors of four other countries.

"The chloride content of Rhine water from the Lake of Constance down to the Dutch-German border rises regularly and sometimes by leaps and bounds," Jaag said, "so that the river carries in its stream some 265 kg of Cl per second, which represents almost 40,000 tons of kitchen salt every day." An enterprising economist might use the Rhine as a sort of economic index; its salt load falls during wars and depressions.

The fact that the Netherlands must draw its drinking and agricultural water from the Rhine, Jaag said, is a "threat to the very life of the Dutch population."

"Waterborne infectious diseases such as outbursts of typhoid and paratyphoid fever occurring here and there in recent times show us that the dreaded epidemics which used to kill hundreds of thousands of people in the old days have not yet been discarded and can be kept in check only so long as the drinking water is given all the attention it deserves."

ACHEMA's abundant program and thousands of instrumental displays were evidence of Europe's renewed scientific and technological vigor. They were also evidence of a kind of bedrock internationalism older than Marco Polo: what works best sells best.

—T. L. CAMPBELL

Note

This report was made possible by the assistance of all the participants whose work is summarized. Karl Kammermeyer, Department of Chemical Engineering, University of Iowa, was general consultant. The photographs on pages 1026–1030 are by Karl Boese, Frankfurt. The photographs on page 1031 were provided by the manufacturer.