The 1981 Pittsburgh Conference: A Special Instrument Report

1981 was the year of the cathode-ray tube at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. The CRT appeared on a wide variety of instruments at the 32nd edition of this venerable show as virtually every type of instrument became more highly automated—reflecting, says one manufacturer's representative, the need to make more efficient use of the $45,000 yearly investment represented by each laboratory technician. Everything else about the meeting was bigger this year also. Attendance grew 7.6 percent to 17,258, the number of exhibitors rose 9 percent to 498, the number of booths at which products were displayed rose 13.6 percent to 1200, and the number of technical papers went up 12 percent to 913. A better indication of the meeting's recent rapid growth are the corresponding figures from 1976—10,500 attendees, 324 exhibitors, and 497 technical papers. The meeting is definitely becoming one of the largest of its type, and that is becoming a problem for Atlantic City, its host for the second time and the scheduled host for 1982. Already, a portion of the famed boardwalk in front of Convention Hall has had to be cordoned off for storage of packing crates because the normal storage space within the hall itself was occupied by the meeting, and future meetings may require more drastic measures. There were some conflicting opinions about whether the increased attendance would be reflected in increased sales leads. One salesman noted the "conventional wisdom" that there are only about 5000 people walking around the meeting with money in their pocket, no matter how many come through the door. Academic scientists were quite obviously more conservative in their buying plans this year, presumably because of the Reagan Administration's planned slashes in National Science Foundation funding for instruments—especially the elimination of the proposed $75 million program for upgrading large instruments at the major universities. Industrial scientists, in contrast, were thought to be on a buying spree whose ultimate cause may be the recent rise in profits in the petroleum and petrochemical industries. A similar contrast could be observed in the employment center. Job openings in government and academia were virtually nonexistent, while industry filled several bulletin boards with vacant positions. These are clearly both the best of times and the worst of times.

Myriad Ways to Measure Small Particles

Most everyone has probably seen those toys that allow children to draw pictures or write messages on a plastic screen with the aid of a magnet that traces out the desired pattern. A would-be manufacturer of one such device, having worked out the details in a prototype, set about producing the sketching toy in volume. But, mysteriously, none of the mass-produced items worked properly. After numerous chemical tests failed to uncover the cause of the malfunction, one of the analytical labs suggested measuring the size of the small, iron particles that were the active agent in the writing medium. By some mischance, according to Richard Karuhn of Particle Data Labs, the Elmhurst, Illinois company that did the measurements, the particles in the production toy were some 5 micrometers smaller, on the average, than those in the prototype. Because of the smaller size, the electrostatic force holding the iron particles to the screen increased to the point that the force from the magnet that ordinarily caused them to move and thereby form patterns on the screen was ineffective.

Underwhelming, you say? Well, perhaps, but the application of physical and chemical principles in the form of instruments that can help solve such apparently mundane problems is what the Pittsburgh Conference is mainly about. However, it is only in the last decade that U.S. manufacturers have realized how important the properties of fine particles can be to a successful product. Applications of instruments for counting and sizing small particles range from controlling the sweetness of chocolate, to ensuring that rocket propellants burn at the right speed, to validating the number of viable sperm in cryogenically frozen semen in animal husbandry. Karuhn predicts that a rapidly growing interest in particle technology will lead to a big demand for people trained in this field. There may also be more sophisticated instrumentation in the not too distant future.

This is not to imply that such instrumentation now lacks the amenities of other kinds of equipment. Automated machines with microcomputers and data systems are available. And there are lasers. Lasers have yet to make much of a dent in the commercial spectrometer market, at least as gauged by the wares exhibited at the Pittsburgh Conference. But these modern-day light sources are already staple items in particle-measuring devices. The way to use lasers that might most stir the heart of a frontier researcher is in photon correlation spectroscopy. A relatively new method for determining the size and diffusion coefficient of particles dispersed in a liquid, photon correlation spectroscopy has not yet reached the status of a mainline technique. One limitation is that it gives only an average particle size rather than a distribution—that is, the number of particles in each size range.

Particles in a liquid are in constant, random motion (Brownian motion). Because of this, laser light scattered by the particles will fluctuate as the phases of scattering contributions from various particles change relative to one another. Moreover, the motion of the particles will be affected by their size, with smaller particles moving faster, and so on. A low-power helium-neon gas laser, which emits red light, is the source. A photomultiplier records the fluctuations in the scattered light. And a special-purpose computer calculates a mathematical entity called a correlation function. The diffusion coefficients and sizes of the parti-

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cles in the liquid are, in turn, extractable from the correlation function.

At the Pittsburgh Conference, Malvern Instruments Ltd. of Malvern, United Kingdom, exhibited an instrument that can measure particles in the size range 30 angstroms to 3 micrometers. It has a capability of measuring scattering at many angles, and the sample temperature can be varied. The cost, including a general-purpose microcomputer in addition to the correlation function signal processor, is $25,000. Langley-Ford Instruments, Amherst, Massachusetts, had on display a photon correlation device covering the size range 50 angstroms to 5 micrometers. Scattering can be measured at only one angle (90°) and only at room temperature. Prices range from $13,400 to $17,800, depending on whether microcomputer control is wanted, plus the cost of accessories.

In addition, Chromatix of Sunnyvale, California, advertised a laser light-scattering photometer with a photon correlation spectroscopy capability. The instrument, which costs $47,700, can measure particles having diameters from 30 angstroms to a few micrometers. Finally, Coulter Electronics, Inc., of Hialeah, Florida, showed its Nanosizer, a microcomputer-controlled photon correlation instrument for measuring particles with sizes from 400 angstroms to 3 micrometers. The Nanosizer price is $24,500.

A more classical use of lasers in particle-measuring instruments is as a source for forward light scattering. The deflection of the light depends on the size of the particles, with larger particles scattering the light through smaller angles and smaller particles through larger angles. The technique cannot, however, reach as small a particle size as photon correlation spectroscopy. The Leeds and Northrup Company of Largo, Florida, for example, exhibited for the first time at the Pittsburgh Conference three instruments that cover different size ranges. All three are microcomputer controlled and can handle high concentrations of particles dispersed in flowing liquids or in airstreams. They are intended primarily for on-line process control. The basic model measures particles in the size range 2 to 200 micrometers and costs $25,000. A second model is adapted to accurate measurement of light scattered through smaller angles. It covers the range 44 to 1000 micrometers and is priced at $35,000. The third model contains a halogen, ultraviolet light source as well as a helium-neon laser to measure small particles scattering light through large angles. The size range is 0.1 to 20 micrometers and the cost is $35,000.

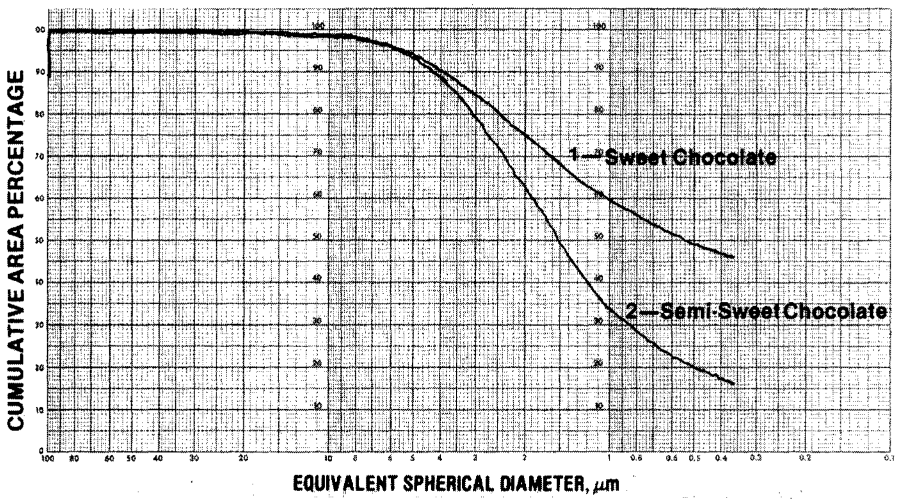
Other manufacturers offering instruments for particle measurement by forward scattering of laser light were Malvern and Spectrex Corporation of Redwood City, California. Malvern's microcomputer-controlled machine covers the size range 1.5 to 500 micrometers with an optional extension to 1800 micrometers and can handle particles in aerosols and sprays as well as those dispersed in liquids or in airstreams. It costs $33,000. The Spectrex instrument is designed for particles in liquids in sealed bottles. The range of particle diameters is 1 to 100 micrometers and the cost is $15,000, including a microcomputer.

It is, of course, possible to count and size particles without recourse to a laser. Two firms that offered optical particle-measuring instruments that did not use lasers were Polytec Optronics, Inc., El Toro, California, and the Hiac/Royco Instruments Division of Pacific Scientific, Menlo Park, California. From pulses of light scattered 90° out of a beam of white light from a halogen lamp, the Polytec instrument can determine the size and velocity, as well as the number of scattering particles in gases or liquids. The device, which can measure particles larger than 0.35 micrometer, costs from $25,000 to $30,000, including a pulse analyzer.

Hiac/Royco takes the opposite tack and measures particle sizes by the decrease in transmitted light when individual particles 0.3 micrometer in diameter or larger in liquids or 0.1 micrometer in airstreams block the light. The company makes two types of instruments. The first monitors unwanted contaminants in nominally pure liquids. The second is designed to look at "wanted" rather than "unwanted" particles. A manufacturer of a metal powder, for example, might want to know the number of particles in various size ranges in the powder. The Hiac/Royco instruments range in price from $9,000 to $35,000, the latter for a computerized machine.

Micro Pure Systems, Inc., of Smithfield, Rhode Island, offered an instrument that uses beams of ultrasound rather than light to detect particles. The system is designed to continuously monitor particles for on-line process control without diverting any material for off-line analysis as most other techniques require. Ultrasonic waves can also penetrate liquids that are opaque to visible light, but they cannot propagate in gases. Prices range from $23,500 for a basic system to $36,500 for an instrument that can detect particles as small as 1 micrometer in diameter. The Micro Pure particle detector can also handle samples from six different points in a process stream at once.

One of the more venerable techniques for sizing particles is sedimentation. A viscous liquid containing particles of varying sizes is the starting point. With time, the larger particles first and later the smaller ones settle to the bottom of the container. There is a mathematical



Particle size analysis of chocolate

These data, taken by the photosedimentation method, show that the sweetness of chocolate is controlled by the size of cocoa particles. Sweet chocolate has more small particles than semi-sweet. [Source: Micromeritics Instrument Corporation]

formula that describes the process. As they settle, the particles block a collimated x-ray or white light beam. Electronic circuitry continuously executes the operations prescribed by the mathematical formula as the particles settle and less and less light is blocked. In the end, a graph giving the fraction of particles at each particle size is generated. In the earliest versions of such instruments, the time required for this analysis could be several hours or even days. Nowadays, the collimated light beam is programmed to scan from the bottom to the top of the container, thereby reducing the time to a half hour or less. Jack Smithwick of Micromeritics Instrument Corporation, Norcross, Georgia, cited as one example of the timesaving possible with automated machines the analysis of particle size in clay. Where it once took 200 hours to track 0.25-micrometer clay particles, it now requires only 67 minutes.

Two companies, Micromeritics and Microscal Ltd. of London, were at the Pittsburgh Conference offering sedimentation instruments. The Micromeritics x-ray version handles particles in the size range 0.1 to 100 micrometers and costs $20,250. The company also manufactures a similar instrument based on the blockage of a visible light beam by settling particles that are transparent to x-rays. This machine covers the same size range and costs $14,950. The Microscal Photosedimentometer, which uses visible light, is designed for particles from 2 to 75 micrometers. Prices start at $5500 for a nonscanning version, the least expensive of three models. Microscal also makes a scanning x-ray instrument that measures particles having diameters from 1 to 75 micrometers and that costs $23,000.

A variation of the sedimentation method is the use of a centrifuge. Two firms exhibiting particle-sizing instruments based on centrifuging were Joyce-Loebl of Marquisway in the United Kingdom and Shimadzu Scientific Instruments, Inc., in Columbia, Maryland. Joyce-Loebl's device covers particles with sizes from 100 angstroms to 60 micrometers and costs $35,000. A data analysis terminal is also available. The Shimadzu instrument measures particles with diameters from 0.1 to 150 micrometers and is priced at $9000. Microscal did not show it at the conference, but the firm does offer a centrifuging x-ray sedimentometer that can measure particles as small as 100 angstroms in size.

Another well-established particle measurement technique does not rely on optical phenomena. Particles are dispersed in an electrolyte containing two electrodes. Between the two electrodes is an orifice. The resistance to current flowing in the electrochemical cell formed by this system depends in part on what is happening at the orifice. In particular, if a particle fills the orifice and displaces some of the electrolyte, the resistance is momentarily increased. The volume of the particle in the orifice, and hence its size, can be calculated from the change in the resistance.

Two companies exhibited particle measuring instruments of this type. One is Particle Data, Inc., Elmhurst, Illinois, whose device measures particles having sizes from 0.3 to 450 micrometers. The company sells four versions of its instrument ranging in price from $9,000 for a manual system to $30,000 for a highly automated one. The second manufacturer is Coulter, whose Coulter Counter is

(Continued on page 152)

Rough Going for Lasers at the Pittsburgh Conference

Lasers have never been a big hit at the Pittsburgh Conference. In part, this may be because the emphasis there has been on instruments that are as rugged and inexpensive as possible and, above all, can be operated by relatively unskilled technicians. Most lasers fit none of these categories. The J-Y Optical Systems Division of Instruments SA, Inc., for example, has exhibited for several years a Raman scattering spectrometer, an instrument that is practical only with a laser as the light source, but J-Y sales representatives have told *Science* that this equipment has never generated much interest. Well-known laser manufacturers, when they come to the conference at all, display other lines of instruments, such as chromatography equipment. Probably the biggest use of lasers has come in instruments that measure the size of minute particles by light scattering (see story, p. 146). These devices use helium-neon gas lasers that are rugged and inexpensive, and for the most part the operator does not need to know whether there is a laser or a light bulb in the machine. This is called making the laser transparent to the user.

Transparency may be the key to lasers penetrating the analytical chemistry market. One company that is trying this approach is the Laser Analytics Division of Spectra-Physics. Several years ago Laser Analytics developed a solid-state infrared laser whose wavelength could be varied by adjusting its temperature within the range from 15 to 100 K. Three such Laser Analytics introduced a microcomputer-controlled isotope ratio company has been marketing a complete infrared spectrometer system built around these sources. This year, Laser Analytics introduced a microcomputer-controlled isotope ratio measurement system that is based on measuring the absorption of light from a tunable solid-state laser at two different wavelengths. A six-page brochure mentions the word laser only four times and emphasizes that a technician can operate the instrument.

The device is intended to measure

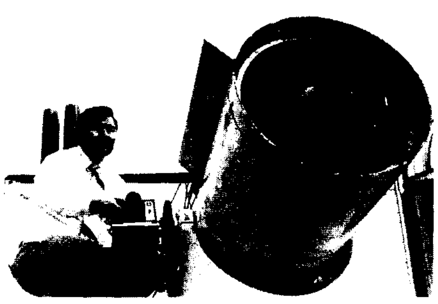
(Continued from page 148)
familiar to a generation of hematology laboratory analysts. The Coulter Counter, which measures particles in the range 0.3 to 1600 micrometers, costs from $16,800 to $30,300, depending on options. This year, Coulter is also offering a microcomputer data system programmed specifically for particle analysis for $6800.

Finally, Micromeritics displayed a prototype of a quite different sort of particle-measuring instrument that the company calls a hydrodynamic chromatograph. The machine is based on a concept developed by Hamish Small of Dow Chemical Company. Like a chromatograph, the instrument has a column stuffed with a nonporous packing. In the column, large particles tend to flow through the center and emerge early, whereas small particles gravitate toward the edge, are retarded, and emerge late. An ultraviolet detector senses the emerging particles. The instrument, designed to handle a wide variety of materials consisting of particles from less than 250 angstroms up to 1 micrometer, is computerized with operating programs residing on floppy disks. A data terminal is also included in the $47,500 price tag that is expected when deliveries begin next year.—ARTHUR L. ROBINSON

New Ways to Measure SO_2 Remotely

Remote measurement of pollutant concentration is a problem of continuing interest to environmental authorities, who frequently are not able either to gain access to industrial facilities or to install monitoring devices on tall smokestacks. Two new devices make it possible to measure the concentration of one important pollutant, sulfur dioxide, in the smokestack plume without ever setting foot inside the factory.

The first is a laser-based instrument developed at SRI International of Menlo Park, California, by a team headed by James Hawley. The instrument is a differential absorption lidar, or DIAL for short; lidar stands for light detection and ranging. The heart of the instrument is a pair of ultraviolet lasers operating at wavelengths in the region of 3000 angstroms. The primary laser operates at a wavelength where sulfur dioxide has a strong absorption band; the second operates at a wavelength 5 angstroms from the first, where sulfur dioxide absorbs only weakly. Each laser is pulsed independently, and the amount of light scattered back toward the laser by the atmosphere is measured with a sensitive detector. Since absorption of light by sulfur dioxide reduces the amount scattered back to the detector, the difference in intensity between the sample and reference beams is proportional to the concentration of the pollutant. The ranging capability of the instrument, which measures the time elapsed between emission of light by the laser and the return of light to the detector, makes it possible to determine the concentration of sulfur dioxide at various points within the plume.



James Hawley

of SRI uses a small laser to aim DIAL at a precise portion of a smokestack plume.

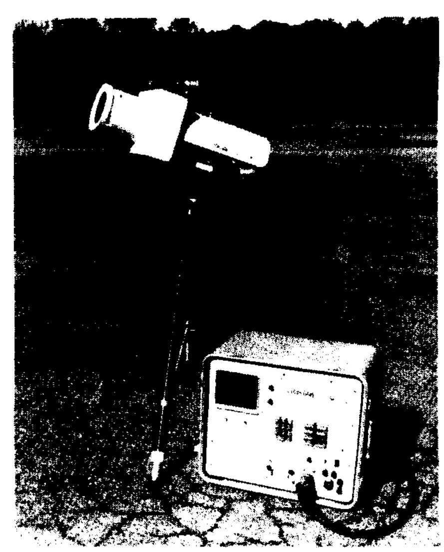
The sensitivity of DIAL is about 2 parts per million·meters (ppm·m) for a 2-minute integration; that means that if a smokestack plume is 10 meters in diameter, the instrument can detect a concentration of 200 parts per billion. Actual concentrations of sulfur dioxide in emissions are generally much higher. Measurements can be obtained when the device is as far as 3 kilometers from the smokestack. If used to make measurements over larger areas, the instrument can also, for example, produce data on the distribution of pollutant gases trapped in temperature inversions.

DIAL is mounted on a 12-meter-long trailer and is powered by a mobile generator. The SRI group recently towed DIAL to Springfield, Illinois, to monitor emissions from the Commonwealth Edison Company's Kincaid Generating Station where they observed a good correlation between concentrations measured with DIAL and those obtained from instruments attached directly to the stacks. But DIAL also obtained information about the dispersal of the plume that otherwise could have been obtained only with a very large number of ground-based air-sampling stations. The instrument has also been used in California to measure emissions from an oil refinery and from several gas- and oil-fired power plants.

The present device, which cost more than $200,000 to construct, is used to perform contract monitoring services for utilities, regulatory agencies, and other groups. If the demand for such service is strong enough, adds Hawley, one or more additional devices could be constructed.

The second new instrument, displayed for the first time at the Pittsburgh Conference, is, at $32,700, much less expensive, but it is also less versatile. Called the Visiplume 121, the device depends on technology originally developed at the NASA–Langley Research Center by Reginald J. Exton and Raymond Gregory. They subsequently left NASA and founded Research Ventures, Inc., of Williamsburg, Virginia, to commercialize the device. Much of the correlation technology used in the instrument was provided by Langley-Ford Instruments of Amherst, Massachusetts, and both companies now sell it.

Visiplume is also based on ultraviolet absorption measurements near 3000 angstroms but it uses the sun as its light



The Visiplume 121