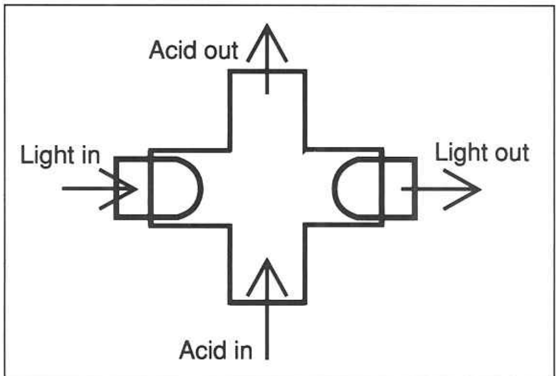
The Optical High-Acidity Sensor

Inventors: Betty Jorgensen, Materials Science and Technology Division; Howard Nekimken, Nuclear Materials Technology Division; and Patrick Carey*



The optical high-acidity sensor quantifies acid concentrations below a pH of 0 rapidly and accurately, while generating no waste.

Mining, metal, and many chemical industries generate large amounts of highly acidic waste. For efficient processing, for economical recycling of acids, and for reduction in the amount of waste discharged to the environment, these industries need an accurate, on-line method of measuring acid concentrations. Researchers at Los Alamos National Laboratory have invented a computer-controlled instrument for taking such measurements, the Optical High-Acidity Sensor. In harsh chemical environments it is faster, simpler, and more effective than any other technique on the market for measuring acidity.

The sensor can function in solutions of high acidity that until now had to be measured by sampling and titration, a cumbersome, time-consuming process that generates caustic waste. The new sensor itself produces no waste. Used on-line, it will save money and reduce waste by making it possible for companies to make process control and waste-treatment adjustments before acidity fluctuations become a problem. It will eliminate the need to reprocess materials that were incorrectly processed because the acidity was not accurately known. Finally, by providing an economical way to measure the concentration in recycled acid, it will encourage recycling and thus reduce discharge into the environment.

The Invention—Characteristics and Advantages

The high-acidity sensor consists of a flow cell in which two fused-silica lenses are tightly mounted across from one another. Fiber optic cables connect the two lenses to a spectrophotometer. One lens is coated with a sensing material that consists of a polymer (polybenzimidazole) and an indicator (Chromazurol-S). The polymer is chemically bound to the lens, and the indicator is physically entrapped within the polymer. The acidic solution to be measured streams up through the flow cell. Light from a fixed source in the spectrophotometer is collimated by the coated lens and then passes through the acidic solution to the second lens, which refocuses it. The amount of light absorbed by the indicator depends on the solution's acidity. The spectrophotometer reports the indicator's absorption spectrum, which gives a measure of the acidity by reflecting the concentration of hydrogen ions in the solution, the method for measuring acidity.

The optical sensor gets its sensitivity and its unique ability to withstand highly acidic solutions for months at a time from its materials. The indicator, Chromazurol-S, is sensitive to high acid concentrations but not susceptible to metal interferents. Metals are unable to chemically bond with the Chromazurol-S under acidic conditions. The polybenzimidazole polymer binds strongly to the chemically treated glass of the lenses and can withstand long-term contact with strong acid solutions. The polymer's affinity for absorbing water allows hydrogen ions access to the indicator, and its small pores allow hydrogen ions to pass through the indicator while preventing most interfering metals from reaching it.

Because of the characteristics of these materials, the sensor has exceptional capabilities. It can measure solutions with a negative pH, whereas techniques using pH electrodes are not suited to monitoring chemical processes in a high-acid range and can measure only solutions that have a pH of 1 or more. The sensor can measure acid concentrations in the 4 to 12 molar range, an achievement until now only possible with sampling and titration. With further development the sensor is expected to have a range of sensitivity of from 0.1 to 12 molar and perhaps up to 16 molar, a range well beyond present-day commercial sensors. The sensor is also stable; it can function in highly acidic solutions for four to six months; and it needs to be calibrated less than once a week. Another advantage is that it is chemically reversible, that is, reusable in several solutions. For all these reasons the Optical High-Acidity Sensor won a 1991 R&D 100 award. The award is given by *Research and Development Magazine* to the one hundred most significant technical innovations of the year.

Applications

The Department of Energy has a patent pending on the Optical High-Acidity Sensor. The principal use for

**Patrick Carey worked on the Optical High-Acidity Sensor while he was a postdoctoral fellow at the Laboratory.*

For more information about the Optical High-Acidity Sensor, please contact Dr. Ronald E. Barks, Industrial Applications Office, Los Alamos National Laboratory, P.O. Box 1663, Mail Stop M899, Los Alamos, NM 87545, Telephone (505) 667-3839.

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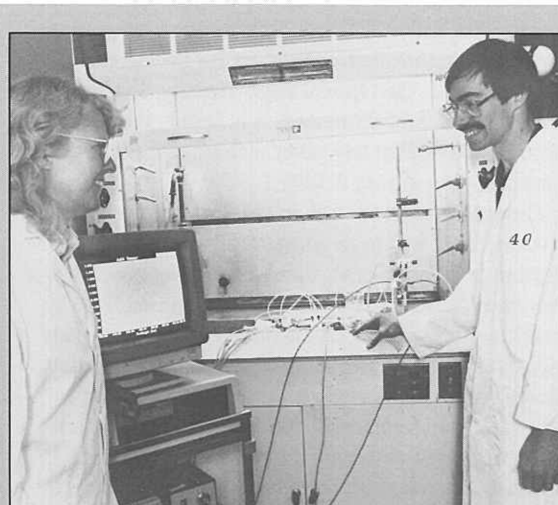
the sensor will be in monitoring highly acidic chemical processes and waste solutions. High-acidity processing is common in industry. Examples are processing metals from mining operations, reprocessing fuel elements from nuclear power plants, and metal-finishing processing like electroplating. The new sensor will improve efficiencies and reduce costs by giving accurate, inexpensive measurements of acidity during processing. In addition, it generates no waste. The sensor, already installed in the plutonium processing line at Los Alamos, can eliminate human error in acid production because data acquisition is computer-controlled.

The researchers are now working on ways to make the sensor compatible with many solvents for a range of other applications. For use in organic chemical production, for example, the sensor must be compatible with a wide variety of organic reagents. In the semiconductor industry, it must withstand high fluoride concentrations, and, because fluoride etches glass, a protective coating for the lenses may be required. For work with chemicals that have high absorptivities in the sensor's spectral absorption band, the researchers are examining spectral subtraction methods. They are also testing the ruggedness of a sensor that has the sensing material coated onto an optical fiber probe for in-line measurements.

The researchers predict that the sensor will be a vital tool for monitoring any highly acidic chemical-processing or laboratory operation. It will save companies thousands of dollars by improving process efficiencies and reducing the time devoted to taking acid measurements. The environment will benefit because not only will more efficient processes produce less waste, but the sensor will also make possible cheaper and easier recycling of the used acids. The result will be a reduction in the amount of acidic waste discharged into the environment.

Inventor Betty Jorgensen, a staff member in the polymer science section of the Laboratory's Materials Science and Technology Division, has research interests in polymer membranes, chelating polymers, and chemical sensors. She began her career as an electron microscopist in a neurobiology research laboratory at the University of California in San Diego, where she had earned a B.S. degree in biology. She came to Los Alamos in 1975, working at first in target fabrication for laser fusion, then in polymer science, and later in the development of process sensors. While working at the Laboratory she took courses in polymer chemistry, photochemistry, and physical chemistry at the University of New Mexico. When a nuclear materials group at the Laboratory needed an acid sensor for plutonium processing, she began work on the Optical High-Acidity Sensor.

Inventor Howard Nekimken, a staff member in the Nuclear Materials Technology Division, develops computer hardware and software for process control for a Laboratory research group that explores new methods of processing plutonium. With a B.S. in chemistry from the University of Wisconsin at Green Bay and a Ph.D. in analytical chemistry from the University of Illinois, he came to the Laboratory as a postdoctoral fellow in 1985 and became a staff member in 1988. His interests are laboratory automation and robotics, chemical sensors, and process control by computer.



Betty Jorgensen and Howard Nekimken work on their Optical High-Acidity Sensor.

Patrick Carey did sensor research on the fiber optics for the Optical High-Acidity Sensor while he was a postdoctoral fellow at the Laboratory from 1987 to 1989. Before that, he earned his B.S. in chemistry at Western Washington University and his Ph.D. at the University of Washington. He is now a postdoctoral fellow at the Center for Process Analytical Chemistry, a National Science Foundation-industry-university cooperative at the University of Washington. His research interests are chemometrics, that is, chemical data analysis, and chemical microsensors.

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