

Science & Technology

REVIEW

July/August 2000



U.S. Department of Energy's
Lawrence Livermore
National Laboratory

Subcritical Explosions Yield New Knowledge

Also in this issue:

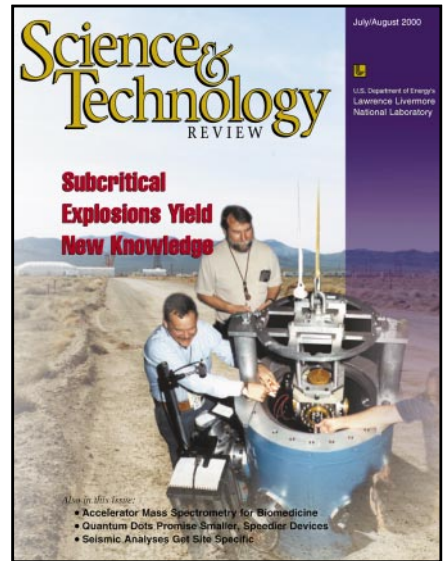
- **Accelerator Mass Spectrometry for Biomedicine**
- **Quantum Dots Promise Smaller, Speedier Devices**
- **Seismic Analyses Get Site Specific**



About the Cover

Project staff inspect an expendable detonation vessel used for Lawrence Livermore's subcritical experiments at the Nevada Test Site. At the center of the steel vessel is a "cube" containing plutonium and chemical explosives. Visible in the photo are (left) Walter Dekin, mechanical project engineer, and Dave Crozier, technician.

The article beginning on p. 4 describes the experiments, including the design and testing of the heavily instrumented vessels that completely contain all detonation products and allow faster, cheaper experiments without compromising safety.



About the Review

Lawrence Livermore National Laboratory is operated by the University of California for the Department of Energy. At Livermore, we focus science and technology on assuring our nation's security. We also apply that expertise to solve other important national problems in energy, bioscience, and the environment. *Science & Technology Review* is published 10 times a year to communicate, to a broad audience, the Laboratory's scientific and technological accomplishments in fulfilling its primary missions. The publication's goal is to help readers understand these accomplishments and appreciate their value to the individual citizen, the nation, and the world.

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Contents

Features

3 Subcritical Tests Are Important to Stockpile Stewardship

Commentary by Michael Anastasio

4 Underground Explosions Are Music to Their Ears

Deep underground at the Nevada Test Site, Livermore scientists are conducting experiments to obtain information on plutonium and its alloys and how their properties change over time.

12 Biomedical Research Benefits from Counting Small

Accelerator mass spectrometry is on its way to becoming a routine tool in biomedical research.



Research Highlight

20 Mighty Small Dots

Departments

2 The Laboratory in the News

22 Patents and Awards

Abstracts

A Web site for tracking water contamination

Once, it was disparate information stored in different places, hard to find and use. Now, after being cataloged, combined, and integrated into a database by Lawrence Livermore scientists, the information has become a Web site called GeoTracker (<http://geotracker.llnl.gov>), a tool for identifying leaking underground fuel tanks and their proximity to municipal water wells. It will help regulators and the public to evaluate the safety of their drinking water.

GeoTracker resulted from concerns over groundwater contamination caused by the gasoline additive MTBE (methyl tertiary-butyl ether). A potential carcinogen that has been discovered to be leaking out of underground fuel tanks and seeping into groundwater, MTBE is slated to be banned statewide in 2002. In the meantime, studies are being conducted to understand its environmental and human health effects, among them, how and where the additive has been transported in the environment.

GeoTracker was mandated by 1997 legislation and made a reality through the efforts of California Assemblywoman Sheila Kuehl of Santa Monica. She was instrumental in getting funds to develop it after city officials determined that there was a leaking underground storage tank next to Santa Monica's municipal water supply.

At the Laboratory, the Web site was developed under project director Anne Happel, a member of the Environmental Protection Agency's blue ribbon panel studying MTBE. The site shows a state map on which a user can zoom in and click on an area for detailed information, including data on locations of underground fuel tanks, tank owners, location and capacity of water wells, how many people are served by those water wells, and other chemicals in the fuels, particularly those thought to be cancer-causing. Laboratory staff spent more than a year cataloging the necessary information. They will run the site for a year and then hand it over to the state.

Site creators say that GeoTracker will give regulators the widest range of information available as well as improve communication between agencies and make information-gathering more efficient. James Giannopoulos, a manager at the State Water Resources Control Board, says that the state is monitoring at least 18,000 cases of leaking underground tanks, 10,000 of which involve MTBE. He adds, "I think [GeoTracker] is going to represent a major change in the way we collect, store, and analyze data."

Contact: Anne Happel (925) 422-1425 (happel1@llnl.gov).

"Superbug" genome decoded

Researchers at the Joint Genome Institute (JGI) in Walnut Creek, California, have completed the first phase of genome sequencing (called the shotgun sequencing phase) for *Enterococcus faecium*, a microbe that is a leading cause of hospital-acquired infection in the United States. It is dubbed a "superbug" because it has a propensity to quickly develop resistance to any antibiotic used against it.

The sequencing work was a collaboration between JGI, led by Elbert Branscomb and Trevor Hawkins, and George Weinstock and Barbara Murray of the University of Texas Health Science Center and Baylor College of Medicine in Houston. The sequencing is notable for the speed at which it was done. An entire strand of *E. faecium*'s DNA, consisting of 2.8 million base pairs, was done in the equivalent of a single day. Says Branscomb, "I believe this kind of a fast response capability could prove to be very useful to researchers in medical, national security, and agricultural contexts."

The work paves the way for researchers to develop a preventive vaccine and better diagnostic tests and treatments to combat the microbe. *E. faecium* is likely to infect only those patients with long hospital stays who are on multiple antibiotics and have a number of medical problems. What is particularly worrisome about the microbe is its adeptness at developing drug resistance. Researchers think it's just a matter of time before the microbe transfers its drug-resistant genes to other, more virulent types of drug-resistant bacteria. "The increase in resistance is a grave signal of the reduced effectiveness of antibiotics," says Weinstock. "If *Enterococcus* passes its resistance to staph, we're in trouble."

Future work at JGI and the Baylor College of Medicine will complete the final assembly of the *E. faecium* genome and provide a more complete analysis of its genetic structure.

The JGI is a merging of the genome programs of Lawrence Livermore, Lawrence Berkeley, and Los Alamos national laboratories.

Contact: Elbert Branscomb (925) 296-5700 (branscomb1@llnl.gov).



Subcritical Tests Are Important to Stockpile Stewardship

ASSURING the safety and reliability of the nation's nuclear weapons stockpile without underground nuclear testing is a formidable scientific and engineering challenge for the Department of Energy and its national security laboratories. For the past three years, a team of Lawrence Livermore men and women has been working in an underground facility at the DOE's Nevada Test Site to help meet that challenge.

In a complex of mined tunnels with instrumentation rooms and experimental alcoves, the team conducts experiments involving chemical high explosives and weapons-grade plutonium. Results from the experiments are playing an increasingly important role in DOE's Stockpile Stewardship Program.

We call the experiments subcritical because the amount of plutonium used in each experiment is so small it cannot reach critical conditions. No self-sustaining nuclear fission chain reaction, like that in the detonation of a nuclear warhead, can occur. We use the experiments to better understand the nature of plutonium and its alloys by strongly shocking them with explosives. The shock is intended to reproduce the tremendous pressures and temperatures that occur when a nuclear device is detonated.

The subcritical experiments are revealing important new information about the properties of shocked plutonium. The experimental data are used to refine the advanced computer simulation codes that run on our supercomputers to help predict any problems with the nation's aging nuclear stockpile. The codes reflect what we know about the materials properties of warhead constituents, especially nuclear materials. We can't make accurate predictions about how plutonium will perform in extreme conditions without a more complete understanding of its properties.

In particular, we need to know more about how plutonium changes behavior as it ages over several decades inside a nuclear warhead. Many nuclear weapons in the current stockpile must be maintained far beyond their intended lifetimes. To investigate the consequences of aging, we compare the shocked behavior of newly machined plutonium with that obtained from dismantled warheads that are decades old.

As described in the article beginning on p. 4, a team of experts from Livermore's Engineering and Defense and Nuclear Technologies directorates is responsible for designing and conducting the Livermore subcritical tests.



The experiments are conducted underground at the remote Nevada facility to minimize any possible environmental impact. Underground tests also minimize costs to taxpayers, because they are far less expensive than those conducted in a comparable aboveground facility.

The subcritical experiments benefit from experience gained from a longstanding program of hydrodynamic experiments of mock warheads conducted at Livermore's remote Site 300 test facility. Indeed, most of the instruments we deploy to characterize subcritical experiments in Nevada are identical to those used at Site 300 for looking deep inside an imploding mock warhead. However, the hydrodynamic tests do not use plutonium and so cannot completely answer all of our questions about what takes place in a nuclear device in the microseconds following high-explosive detonation. Only subcritical tests can provide that information.

As we continue our program of subcritical experiments, we're learning how to perform them better, faster, and cheaper. For example, we're currently saving considerable money—and advancing our test schedule—by conducting experiments in steel cylindrical vessels that act as miniature laboratories to confine the explosive products from each experiment. That innovation, which significantly reduces the need to mine new experimental alcoves, is one of several we have made to the program during the past year.

The results from subcritical tests, combined with the steady progress from other elements of the nation's stockpile stewardship program, are strengthening our confidence that we can continue to keep the nation's stockpile safe and reliable.

■ Michael Anastasio is Associate Director, Defense and Nuclear Technologies.

Underground

In the current era of no nuclear testing,

Explosions

subcritical experiments are helping to safeguard

Are Music to

the nation's nuclear stockpile.

Their Ears

BAGPIPE, Oboe, Clarinet, and Piano could be the elements of an avant-garde musical ensemble. At Lawrence Livermore, however, they are the names of recent explosive tests conducted deep under the Nevada desert by Laboratory physicists and engineers. The tests are aimed at providing important data to experts watching over the nation's nuclear weapon stockpile.

In the Livermore experiments, chemical high explosives are detonated next to samples of weapons-grade plutonium (plutonium-239) to obtain new insights about plutonium and its alloys in the ensuing microseconds. The tests, conducted at the Department of Energy's 3,500-square-kilometer Nevada Test Site, are subcritical. That is, no critical mass is formed, so no self-sustaining nuclear fission chain reaction occurs as it does in a nuclear detonation. The experiments are permitted within the Comprehensive Test Ban Treaty signed by President Clinton in 1998.

Indeed, subcritical tests have become largely accepted internationally for ensuring the safety and reliability of a nation's nuclear force without resorting to nuclear testing. Russia has been conducting subcritical tests involving both weapons-grade plutonium and uranium since 1995 at its Novaya Zemlya test site near the Arctic Circle.

Lawrence Livermore's heavily instrumented experiments provide data on the behavior of plutonium in a strongly shocked state and how that behavior differs from plutonium that has aged over decades inside a nuclear warhead. "We need to better understand how the aging of plutonium could affect the safety or performance of a weapon," says Livermore physicist Dick Lear. He explains that the accumulation of alpha particles (helium nuclei) produced by the radioactive decay of plutonium atoms is thought to cause imperfections in the material's crystalline structure and thereby possibly affect its performance. To investigate the consequences of aging, subcritical tests compare the behavior of newly machined plutonium with that obtained from old, dismantled warheads.

The tests focus on ejecta and spall, phenomena that are thought to affect the performance of a nuclear warhead, specifically that part of the warhead called the primary. Ejecta are a violent spray of plutonium particles that are propelled from a material's surface when it is compressed by a powerful shock wave. Spall is the breakup of plutonium from the explosive shock wave reflected back from the surface.

Tests Provide Real-World Data

The Livermore tests, together with those performed by Los Alamos National Laboratory, play an important role in DOE's Stockpile Stewardship Program to ensure a safe and reliable nuclear weapons stockpile without underground nuclear testing. Stockpile stewardship depends in great measure on advanced computer simulations of weapon performance and materials aging. Subcritical experiments provide the actual data about the behavior of plutonium and thus are useful for improving computer simulation codes, enabling them to more accurately predict any

problems with the nation's aging stockpile.

The Nevada tests are an important complement to hydrodynamic experiments on mock warheads conducted at Lawrence Livermore's remote Site 300 test facility in California. While similar to subcritical tests, the hydrodynamic experiments do not use plutonium. Because plutonium is the most enigmatic element in the periodic table (see *S&TR*, June 2000, pp. 15–22), tests using its surrogates cannot accurately answer all the questions scientists have about the behavior of plutonium in a warhead.

The Livermore tests are conducted by the Laboratory's Engineering and Defense and Nuclear Technologies directorates with support from Bechtel Nevada Corp. and AlliedSignal/Federal Manufacturing and Technologies. Livermore researchers share their experimental results with their Los Alamos colleagues.

The subcritical tests are conducted at the U1A complex of the Nevada Test Site, located about 140 kilometers northwest of Las Vegas. The complex



The U1A complex at the Nevada Test Site consists of several buildings and instrumentation trailers from which scientists monitor experiments conducted underground.

consists of several buildings and instrumentation trailers from which scientists monitor experiments conducted in tunnels mined some 290 meters underground. According to Lawrence Livermore engineer Dave Conrad, staging subcritical tests underground is ideal because it minimizes the tests' environmental impact. Conrad, who serves as Livermore's test director and project leader, also points out that underground

testing costs far less than designing, building, certifying, and using an aboveground, reusable chamber.

Tunnels Carved Underground

The underground complex consists of several main tunnels (called drifts), each about one-quarter of a kilometer long, and a series of small experimental alcoves branching off from them. The alcoves are also called zero rooms, from the "ground zero" parlance of the nuclear

test era. The downhole environment is surprisingly comfortable, with well-lit rooms, concrete floors, tall ceilings, and lunchrooms.

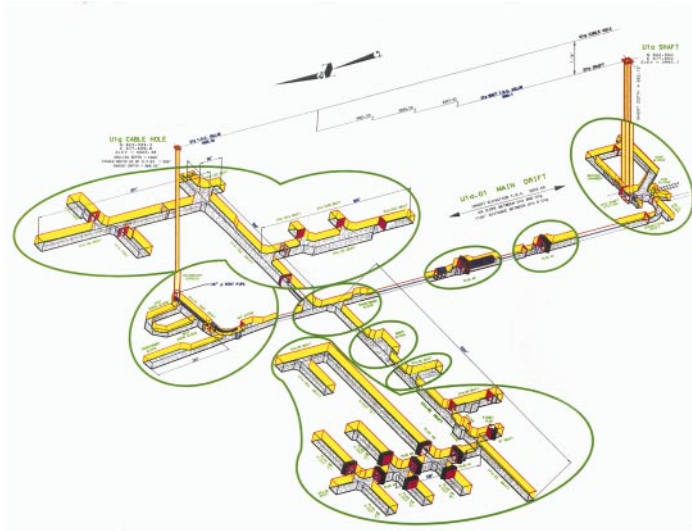
Both Livermore and Los Alamos have designated testing areas in the complex. Los Alamos scientists conduct experiments about every 15 months, while Livermore currently conducts its tests every six weeks, thanks to the use of expendable steel vessels that confine debris from the experiment.

The complex's main vertical shaft is equipped with a mechanical hoist to transport workers and equipment. The shaft was originally mined in 1968 for an underground test that was later canceled. In 1988, the shaft was reopened and a 445-meter horizontal tunnel was mined south of the shaft for a low-yield Los Alamos nuclear test. The test, called Ledoux, was conducted in 1990, two years before President Bush announced a nuclear test moratorium that remains in effect. A second vertical shaft about 305 meters away, constructed in 1991-92, provides cross ventilation, utility access, and emergency egress.

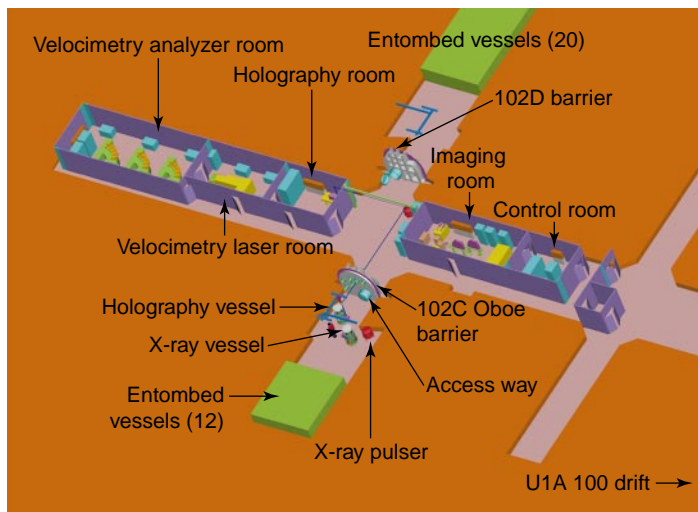
In 1996, Lawrence Livermore started mining its first downhole experimental area, called the 101 drift, using the same mining techniques as those for subway construction. The drift and three small experimental alcoves were completed about 10 months later. The mined areas were stabilized with 5-meter-long steel rods drilled into the tunnel walls, secured with epoxy cement, and sprayed with a slurry of fibercrete, material similar to concrete. The Holog, Bagpipe, and Clarinet test series were all conducted in their assigned alcoves, which afterwards were permanently sealed.

Last year saw the completion of Livermore's second drift, called the 102 minicomplex. Requiring nearly two years to mine, the minicomplex consists

The U1A underground complex has several main tunnels, each about one-quarter of a kilometer long, and a series of experimental areas. Both Lawrence Livermore and Los Alamos have designated areas in which to perform subcritical tests.



Livermore's 102 minicomplex was completed last year. It consists of a suite of diagnostic rooms and two experimental alcoves dedicated to the Oboe and Piano test series.



of a main drift (82 meters long, 7 meters wide, 6 meters high) and two reusable experimental alcoves (23 and 27 meters long; both are 6 meters wide by 5 meters high). The first alcove is planned to accommodate up to 12 Oboe experiments conducted in steel confinement vessels and one Piano experiment that is possibly too large for a vessel. The second alcove is planned to accommodate 20 additional Oboe experiments and another Piano-like experiment.

Holography Provides 3-D View

The high-speed diagnostic instruments used in the experiments are similar to those found in research facilities back at Livermore. The primary diagnostic is a laser-based imaging system that captures the cloud of plutonium ejecta flying out from the shocked surface at the moment of explosion (called shock breakout). The film of this experiment is actually a hologram, which, when projected with a laser, allows experimenters to “walk through” a cloud of plutonium particles in three dimensions. The hologram provides data on the size, shape, number, and velocity of the particles. High-speed cameras also record images of the shocked plutonium over time.

Other instruments, also in regular use at Livermore, complement the holography data on plutonium ejecta. The Fabry-Perot interferometer (see *S&TR*, July 1996, pp. 12–19) examines the change of position (and inferred mass and velocity) of particles trapped on a gold foil by measuring the wavelength shift of laser light reflected from the moving foil surface. Also, the rate at which ejecta hit a set of piezoelectric “pins” is recorded.

Radiography experiments look at another phenomenon, plutonium spall. Like an echo, the shock wave caused by high explosives is reflected back from



Equipment typically used for making subway tunnels was used to carve out Livermore's underground test facilities.



One of two experimental alcoves in the 102 minicomplex during construction. Workers are installing the utilities required to stage and diagnose subcritical tests.

the plutonium surface. Depending upon the shape of the shock wave, the plutonium can develop cracks in its crystalline structure or even begin to break into pieces. Radiography instruments determine the dependence of spall characteristics on shock-wave geometry and changes in pressure.

Currently, the x-ray equipment used on the subcritical tests cannot penetrate materials as deeply as scientists would like. But moving underground a giant machine, such as that at Livermore's Flash X-Ray Facility, is simply not possible, given the cramped shafts and the limited real

estate downhole. Therefore, a Livermore team is building an x-ray machine that will be powerful enough for the tests and small enough to be transported and set up underground.

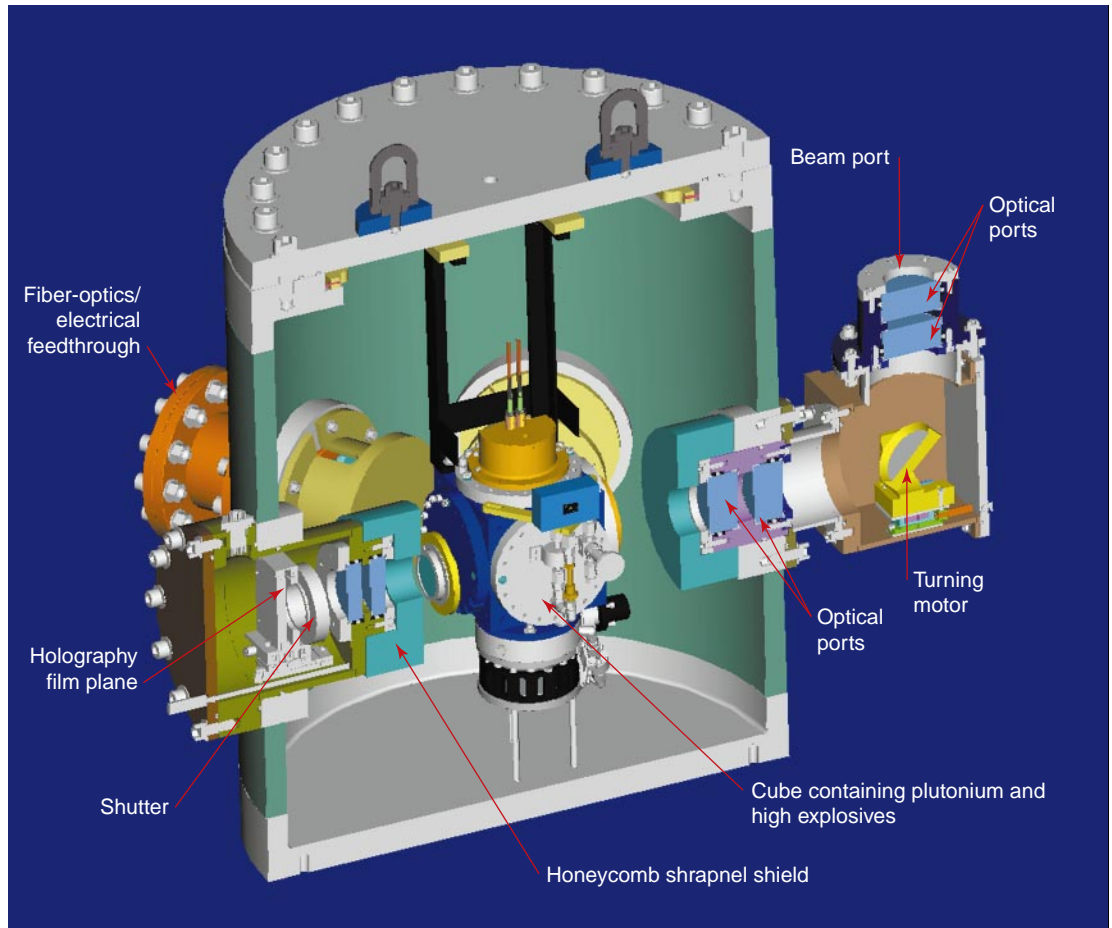
Conrad notes that because of the wide-ranging use of optics in experiments, dust is a major enemy. Unfortunately, dust is a natural attribute of the belowground work environment, especially mining activities. The Livermore team uses a host of techniques to keep dust at bay, including air filtration systems, shoe cleaning machines, and sticky tape on floors.

Holog Test Was First

The first Livermore subcritical test, consisting of two experiments, was conducted in September 1997. It was named Holog, after the major holographic diagnostic technique that was used to examine ejecta. Because Holog was the first experiment, scientists paid special attention to understanding the physics of the explosions and the effects on the containment barrier to the alcove.

One year later, Livermore conducted its second subcritical test. Code-named Bagpipe, this set of four experiments was designed to

This cutaway of a steel vessel shows how laser-based imaging equipment is configured to obtain information on ejecta at the moment of detonation. When illuminated with a laser, the exposed film, actually a hologram, allows experimenters to "walk through" a cloud of plutonium particles in three dimensions.



investigate both ejecta production and spall at different pressure regimes. “Bagpipe confirmed that we can successfully field a broad suite of diagnostics on subcritical tests,” says Lear. “It gave us confidence to design more complex experiments.”

The February 1999 Clarinet test consisted of three experiments that were evaluated by a larger array of diagnostic packages than were used previously. Diagnostic techniques included Fabry-Perot velocimetry, holography, pins, and radiography.

Two of the Clarinet experiments primarily used holography to examine

the effects of aging on plutonium. The two were identical in every way except for the age of the plutonium. The size and distribution of ejecta particles resulting from the shock wave in the two plutonium materials were captured on holographic film and analyzed. From the two holograms, scientists inferred changes due to the aging process.

Oboe Inaugurated Minicomplex

Beginning in September 1999, the Oboe test series inaugurated the new 102 minicomplex, where expendable steel vessels that completely confine the experiments were first used. These steel

vessels make it unnecessary to carve out a separate alcove for each experiment. The alcove is checked for plutonium leakage after an experiment, and if there is no contamination, workers reenter, move the used steel vessel to the rear of the alcove, and entomb it in concrete at least 30 centimeters thick. Preparations then begin on the next experiment.

“We were looking for ways to do the experiments better, faster, and cheaper,” says Conrad. “We asked ourselves if a steel vessel could serve as a miniature zero room, with diagnostic equipment located just outside.”

Underground Tests Earn High Safety Marks

In many ways, subcritical tests at the Nevada Test Site are similar to those that have been conducted safely for years at Lawrence Livermore’s Site 300 research facility. However, Livermore managers have adopted many new safety procedures for the Nevada tests because plutonium has been introduced, testing is being conducted underground, the diagnostic systems are remotely controlled from above ground, and several diverse organizations, each with its own work culture, are involved.

“The underground work environment is congested and there are many potential hazards,” says Livermore test director Dave Conrad. Fortunately, planning for Livermore’s first subcritical tests was begun during the introduction of Integrated Safety Management, the Department of Energy’s program to ensure the highest safety performance. Following the tenets of the safety program required a review of every activity, from mining to experiment setup, before it was undertaken.

The careful preparation has clearly paid off. For example, during the two years of construction activities related to the newest underground minicomplex, called the 102 drift, there were no major injuries or any involving lost time. Safety continues as the highest daily work priority, and quarterly safety walkthroughs are conducted with representatives from the Department of Energy, Lawrence Livermore, and Bechtel Nevada.

A number of safety systems ensures safety to workers and the environment. For example, a computerized safety interlock system of 87 sensors keeps track of the location of workers downhole. Another system continually samples the underground air quality. For added safety, each worker and visitor carries a portable breathing apparatus for emergencies and certain underground areas have been designated emergency shelters.

Plutonium and high explosives are shipped separately from Livermore and then assembled into an experimental package at the Device Assembly Facility located about 16 kilometers from the subcritical test complex (see *S&TR*, May 1998, pp. 23–25). From there, the experimental packages are trucked to the complex and lowered for transport to the experimental alcove.

A major emphasis is on keeping the plutonium used in the tests completely confined. “We take great care that no plutonium will ever leak out into the general environment,” says Conrad. The experiment team’s first commandment, he says, is that “not one atom of plutonium shall be released to any uncontrolled environment as a result of the experiment.”

The containment plan for the current Oboe series of experiments uses the time-tested concept of nested barriers: first there is the expendable steel vessel in which the experiment is conducted; then the experimental alcove, with its locked and temporarily sealed crawl tube; and finally, a large concrete and steel barrier enclosing the drift. Lawrence Livermore safety experts account for the remote possibility that plutonium could seep into the alcove from a crack in the vessel or a seal failure on one of the vessel’s ports. In that case, the alcove would contain any plutonium within the room. The alcove would be in a situation similar to that of the alcoves for the Holog, Bagpipe, or Clarinet tests, which were permanently sealed with concrete following the experiments. Lawrence Livermore managers note that no plutonium has ever been released from an experimental area.

All underground areas and all personnel are monitored for radiation exposure. Workers are not allowed into the reusable alcoves following an experiment until detectors show that there has been zero leakage of plutonium from the experimental steel vessel.



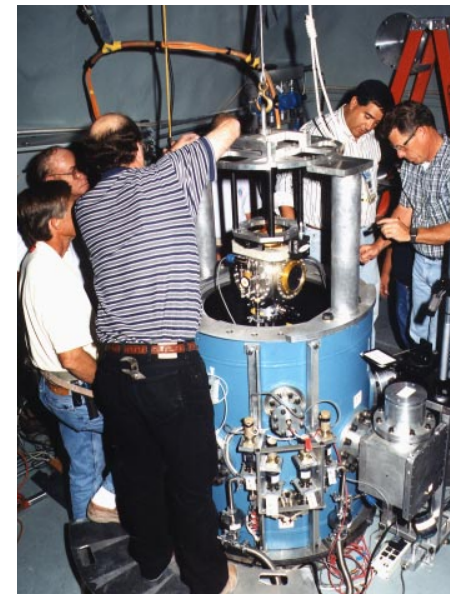
Once lowered downhole, a confinement vessel for the Oboe test series is transported to the experimental alcove. The vessel is designed to confine the gases and radioactive debris generated by a typical experiment.

The vessels with 3-centimeter-thick walls were designed by an engineering team from Lawrence Livermore and Bechtel Nevada. They measure 1.2 meters long and 1 meter in diameter, compact enough to move through the crawl tube of the containment barrier separating the alcove from the corridor. Computer simulations were used to design and test the vessels to ensure that they would reliably confine the detonation products. The design was then experimentally tested at the High Explosives Applications Facility at Lawrence Livermore and the Big Explosives Engineering Facility at the Nevada Test Site. In addition, each newly constructed vessel is pressure-tested to 42 kilograms force per square centimeter, a more-than-adequate pressure to confine the gases and radioactive debris generated by the typical experiment.

Once a new vessel is transported into the alcove, a “cube”—an experimental package containing plutonium and



The steel vessel is pulled through the entryway into the experimental alcove.



Livermore and Bechtel Nevada workers lower the “cube,” which contains plutonium and chemical explosives, into the heavily instrumented vessel.

chemical explosives—is lowered inside. The vessel and the cube are typically maintained at different atmospheric pressures. The vessel is only moderately evacuated; residual air pressure is used to lessen the shock of exploding debris. The cube is at a much lower pressure to permit a laser beam to penetrate the exploding plutonium without being diffracted by air molecules.

Concrete Entombs Vessels

Conrad says the expendable vessels have resulted in significant cost savings and improved data return. “The vessels permit turnarounds faster than anyone had believed possible,” he adds. Oboe experiments, for example, have been performed about every six weeks, at a much lower cost per experiment. “With expendable vessels, we can learn from one shot and apply it to the next experiment just a few weeks away,” he says.

Conrad notes that Holog, Bagpipe, and Clarinet each were fired as clusters of experiments in dedicated alcoves. It took about one year to complete mining activities for the separate alcoves and to set up each experiment cluster, with much of the diagnostic equipment installed in the same room. (Image recording had to rely on sophisticated optical-relay systems connected to equipment outside the alcove.) After the experiment, each alcove and its resident diagnostic equipment were permanently contaminated from the detonation and could not be reused.

By significantly reducing the need for new alcoves, the vessels save mining costs of about \$20 million and double the usable lifetime of the 102 minicomplex from two years to four. Scientists expect to stage up to 12 Oboe experiments in the first alcove of this minicomplex before they start running out of room from the accumulation of entombed vessels. Then, they plan to fire the first Piano shot because it is possibly too

powerful to be confined within a vessel. After that, the alcove will be permanently sealed, as was done with the Holog, Clarinet, and Bagpipe alcoves, and experimenters will move on to the next alcove.

Conrad notes that the new format allows diagnostic data to be recorded either inside or outside of the alcove, depending upon the experimental configuration. Some data may be transmitted over electrical or fiber-optic cables to recording instruments outside the alcove, while other data are recorded inside the room and retrieved following the experiment.

For example, holography film is placed next to the high-resolution lens of a viewing port of the vessel. By locating the film very close to the experiment, scientists have obtained Oboe holograms with unprecedented quality and without the cumbersome optical-relay system used on Holog, Clarinet, and Bagpipe.

Lawrence Livermore physicists have been pleased with the data obtained from subcritical tests and how they are allowing colleagues to

refine advanced simulation codes that run on supercomputers. Lear says experimenters are particularly pleased with how well the expendable vessel concept has worked.

The subcritical tests are envisioned to continue for the foreseeable future as experimenters gain confidence and experience. Mining has begun on an addition, called the 104 minicomplex, to accommodate experiments planned for the middle part of the decade. Those experiments will also be named after musical instruments. Livermore scientists hope these tests will continue to produce data that are music to their ears.

—Arnie Heller

Key Words: Big Explosives Engineering Facility, Device Assembly Facility, ejecta, High Explosives Applications Facility, Integrated Safety Management, Nevada Test Site, plutonium, Site 300, spall, stockpile stewardship, subcritical tests, U1A complex.

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About the Scientist



DAVID C. CONRAD is the test director for the Nevada Experiments and Operations Program. He provides project planning, coordination, and integration of multidisciplinary teams for the execution of tests, projects, demonstrations, and experiments at the Nevada Test Site. He served as the test director on Holog, the first subcritical experiment to be conducted by Lawrence Livermore. Conrad received a B. S. in electronics engineering from Mississippi State University and an M.S. in electronics engineering from the Georgia Institute of Technology. At Lawrence Livermore since 1978, Conrad has had various assignments and managerial positions, including technical coordination of the separator controls for the Atomic Vapor Laser Isotope Separation project and operations management of Livermore’s Industrial Partnerships and Commercialization Program. He has also served as U.S. technical expert for treaty verification negotiations in Geneva and Moscow.

Biomedical Research Benefits

It's no fluke. An experiment that can track 35 micrograms of folic acid through a human for 200 days is routine for Livermore's Center for Accelerator Mass Spectrometry.



Members of Livermore's core biological AMS team stand in front of the new, small spectrometer (also seen in the larger photo) dedicated to biological studies using carbon-14. From left: John Knezovich, Bruce Buchholz, Graham Bench, Ken Turteltaub, John Vogel, Ted Ognibene, and Mark Roberts. Missing from the group is Karen Dingley.

THE short history of accelerator mass spectrometry (AMS) for biomedical research at Livermore has been sweet indeed. Just 10 years ago, Livermore scientists first used AMS to determine how low doses of a suspected carcinogen affect the DNA of mice. A remarkably sensitive measuring technique, AMS can seek out one carbon-14 isotope from among a quadrillion other carbon atoms. It achieved a tenfold improvement in detecting damaged DNA over the best methods then available, thus enabling studies to be conducted directly with humans. Ever since, Livermore has been expanding the development of AMS for biomedical and pharmaceutical applications and is today a recognized leader in the field. (See **box on p. 14** for information on how AMS works.)

Livermore researchers are continuing to study the effects of carcinogens on humans and animals. Perhaps not surprisingly, they have discovered that humans and animals metabolize these substances differently, with resulting differences in the way DNA is affected. AMS is being used in collaborations with researchers from around the world to begin to solve many challenges in biomedicine—from examining the way we metabolize vitamins to developing a new cancer diagnostic test. AMS has even proved invaluable for learning how pesticides move through ant colonies from workers to the queen.

When Livermore proposed that the National Institutes of Health (NIH) establish a National Research Resource for AMS at Livermore, University of California (UC) and Department of Energy scientists provided key testimony to demonstrate the value of AMS. Along with all the other evidence, their testimony must have been persuasive. Last November,

from Counting Small

Lawrence Livermore joined an elite group of research facilities when NIH awarded the five-year grant. There are other NIH Research Resource facilities for various types of mass spectrometry, but this is the first for AMS. An NIH review of Livermore's proposal had this to say:

An overwhelming case was made for the need for an AMS resource and a number of outstanding collaborative projects have already been initiated. . . . At the present time, the LLNL Resource is clearly the most advanced site in the U.S. to explore the use of AMS in biomedical research.

Chemist John Knezovich, director of Livermore's Center for Accelerator Mass Spectrometry (CAMS), is pleased with progress to date. "CAMS is unique in this country in concentrating on biological AMS. Beyond integrating the Laboratory's scientific expertise in both AMS and biomedical research, CAMS provides a major facility that

is enabling research projects from all over the world."

He adds, "A big plus is that in addition to the large, multipurpose AMS machine that we've had for years, a much smaller one will soon come on line that is dedicated to biological studies using carbon-14. And, we have begun to use yet another new spectrometer for biological samples that have been tagged with tritium, a new tracer element for AMS. On top of that, we have added a heavy-isotope line to our large AMS machine for studies of plutonium." (See [box on p. 17](#) for more information on AMS equipment at Livermore.)

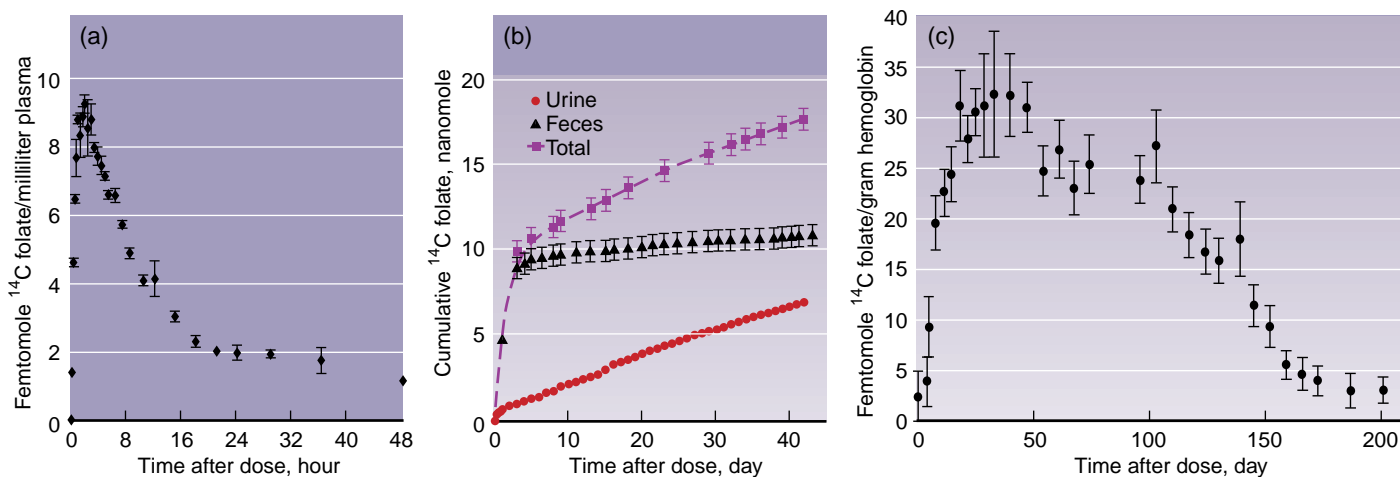
An important aspect of Livermore's work to date has been in establishing AMS as a routine biomedical research tool. An AMS experiment no longer requires a large and expensive staff of physicists and technicians. Livermore scientists have led the technological advances necessary to make AMS a more effective, dependable, and economic tool for the biomedical, pharmaceutical,

chemical, and clinical communities (see also *S&TR*, [November 1997](#), pp. 4–11).

Established in 1989, CAMS was designed to diagnose the fission products of atomic tests; monitor the spread of nuclear weapons to other countries by detecting telltale radioisotopes in air, water, and soil samples; and use isotopic tracers to study climate and geologic records. It still does all these things today—and much, much more.

Sensitivity Is the Key

So what is the value of AMS to you and me? AMS is an ideal method for tracing the passage of chemicals through humans without disturbing normal metabolic processes. Perhaps researchers want to know how the human body metabolizes a drug or vitamin. The molecules of the substance are manipulated slightly to "tag" them with a radioactive isotope, typically carbon-14, though other radioisotopes may be used as well. Rare radioisotopes of elements found in organic materials are used as tracers



A single dose of carbon-14-tagged folic acid was traced for 200 days. (a) The tagged folic acid appears very quickly in plasma (the liquid part of blood) and tapers off in about two days. (b) The amounts of tagged folate being eliminated in feces and urine were followed for 40 days. (c) Folate begins to be incorporated into hemoglobin at day 5. (Hemoglobin is the iron-containing, oxygen-carrying molecule in red blood cells.) The level of folate in hemoglobin peaks at about the 30th day and disappears only after 200 days.

because they can be incorporated into biomolecules and because they are present naturally at low levels, so tagged molecules can be detected easily.

A collection of human subjects swallows or otherwise ingests the substance. Then, using AMS to

measure the number of carbon-14 atoms in samples of urine, feces, saliva, or blood over the next hours, days, and weeks, researchers can trace how much of the substance is absorbed, how it travels through the body, what organs it affects, how much of it is lost through excretion, and so on. The first

experiment to trace the vitamin folic acid in a human followed a single dose of just 35 micrograms for a remarkable 200 days.

Information this detailed has never before been available using healthy human subjects. Less sensitive measuring techniques,

Accelerator Mass Spectrometry at Livermore

Livermore's Center for Accelerator Mass Spectrometry (CAMS) is home to the most versatile and productive AMS facility in the world. AMS is an exceptionally sensitive technique for measuring concentrations of isotopes in small samples, typically less than 1 milligram, and the relative abundance of isotopes at low levels. It can, for example, find one carbon-14 isotope among a quadrillion other carbon atoms.

Mass spectrometry has been used since early in the 1900s to study the chemical makeup of substances. A sample is put into a mass spectrometer, which ionizes it and analyzes the motion of the various ions in an electric field to sort them out by their mass-to-charge ratios. The basic principle is that isotopes of different masses move differently in a given electromagnetic field.

In accelerator mass spectrometry, the same principle applies but the process is different. Negative ions made in an ion source are accelerated in a field of millions of volts. The accelerated ions smash through a thin carbon foil or gas that destroys all molecular species. After passing through a high-energy mass spectrometer and various filters, the ions finally slow to a stop in a gas ionization detector. (See the layout at right of the large AMS machine at Livermore.) The identity of individual ions can be determined from how the ions slow down. For example, carbon-14 slows down more slowly than nitrogen-14, so those ions of the same mass can be distinguished from one another. Once the charges are determined, the detector can tell to which element each ion belongs and counts the

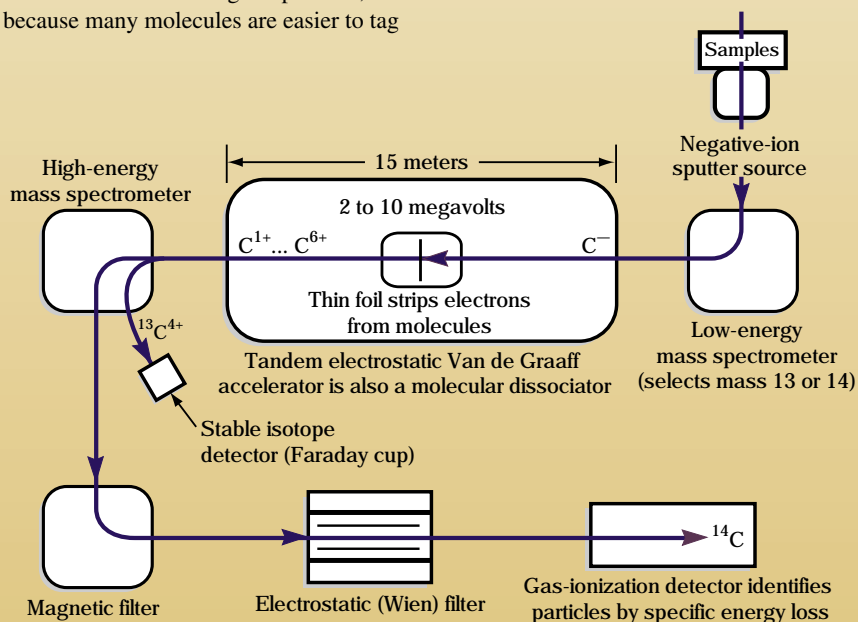
desired isotope as a ratio of a more abundant isotope—carbon-14 as a ratio of carbon-13, for example.

The two "tricks" that make AMS work so well are the molecular dissociation process that occurs in the accelerator and the ion detection at the end. The resulting sensitivity is typically a million times greater than that of conventional isotopic detection.

For biological studies, AMS has been used primarily for counting carbon-14 because carbon is present in most molecules of biological interest and carbon-14 is relatively rare in the biosphere. Tritium (hydrogen-3) has also been used extensively as a tracer in biological research. The use of tritium in AMS is new and holds great promise, because many molecules are easier to tag

with tritium than with carbon-14. Other isotopes are measured by AMS as well, including plutonium-239, calcium-41, beryllium-10, chlorine-36, and iodine-129.

All over the world, AMS is still used primarily to count carbon-14 in archaeological and geologic samples for dating purposes. In the 1980s, it replaced the traditional method of scintillation counting for precise radiocarbon dating, which was time-consuming and required relatively large samples. Livermore performs radiocarbon dating and many other forms of AMS 24 hours a day, 7 days a week for its own research and collaborations as well as for others on a fee-for-service basis.



such as scintillation counting, require ingesting large doses of both the chemical being studied and the radioisotope, something few people want to do. Sometimes, such studies are done with volunteers. Usually, however, scientists have used animals for their research. Then considerable extrapolating has been required: from large doses to small doses and from laboratory animals to humans.

Now researchers can use much smaller, more realistic doses on human subjects. They can measure the true effects of a typical dose of, say, vitamin A or aspirin. AMS, the only method that can trace these low doses over such long time periods, has been described as the most significant new tool for nutritional studies since the 1930s.

And the amount of radioactivity taken in with the chemical being studied is less than one would encounter during a single day's exposure to background ionizing radiation from walking around in the sunshine. (Cosmic rays contain a small amount of radioactivity that we are exposed to every day.) An airplane flight exposes us to far more ionizing radiation than participation in one of these experiments.

Biochemist Ken Turteltaub, one of the developers of AMS for biological applications, is sold on the process. He says, "With accelerator mass spectrometry, we can address problems that cannot be solved otherwise."

Grant Recognizes Achievement

The Livermore team spent five years building and demonstrating the capabilities that led to the NIH grant to Livermore. Today, Turteltaub is the grant's principal investigator, assisted by fellow biochemist Karen Dingley and a team of CAMS scientists. As an NIH Research Resource for biological AMS, Livermore is charged with providing biological researchers throughout the country with access to carbon-14 AMS analysis in their research, developing new methods and instrumentation for the use of AMS in biomedical research, demonstrating new applications, and educating the biomedical research community on AMS. All of these functions have been under way at Livermore for many years, which makes award of the grant particularly gratifying as recognition of a job well done.

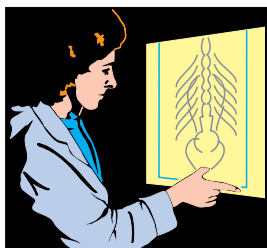
All NIH Research Resource grants focus on collaborative work in order to educate the biosciences community. For

this grant, Turteltaub and his cohorts are working to expand support for the use of AMS and to export its use to as many researchers as possible.

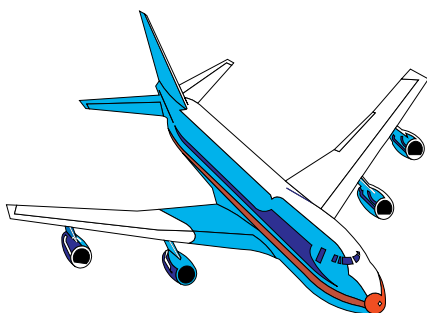
In work that is just beginning, they are also developing new experimental methods so that AMS can be applied to as many types of biological research as possible. They are developing the sample preparation and analytical methods necessary to reduce handling, automate processes, and increase sample throughput. The team will explore new spectrometer components that allow the direct interface of bioanalytical instrumentation to the spectrometer for simplified, rapid, on-line analysis. Finally, further development of the small carbon-14 spectrometer will help to bring AMS machines to more bioanalytical laboratories.

Measuring Damage to Molecules

Turteltaub, Dingley, and other researchers at Livermore are already involved in several collaborations with researchers in the U.S. and England to examine the effects of substances produced by cooking meat. Both 2-amino-1-methyl-6-phenylimidazo [4,5-b]pyridine (PhIP) and 2-amino-3,8-dimethylimidazo[4,5-f]quinoxaline



Chest x ray: 50 microsieverts



1-hour plane flight at 9,000 meters: 5 microsieverts



Typical AMS nutrient study:
1 microsievert



Typical AMS drug or toxin study: 1 nanosievert

Taking a 1-hour plane trip exposes a person to 5,000 times more ionizing radiation than participating in an accelerator mass spectrometry study of a drug or toxin. Everyone on Earth takes in about 2.5 millisieverts of radiation every year. (A sievert is a Système International [SI] unit for radiation dose. It is equal to 8.38 roentgens. One microsievert is one millionth of a sievert and a nanosievert is one billionth of a sievert.)

(MeIQx) are heterocyclic aromatic amines that have been shown to cause cancer in laboratory animals when administered at high doses. The team has used AMS to establish whether DNA and protein adducts (damage) can be detected in laboratory animals and humans when

they take in a smaller, more typical dietary amount of these substances.

In numerous experiments using carbon-14-tagged PhIP and MeIQx molecules, the team has confirmed not only that adducts can be detected at low doses, but also that humans may be

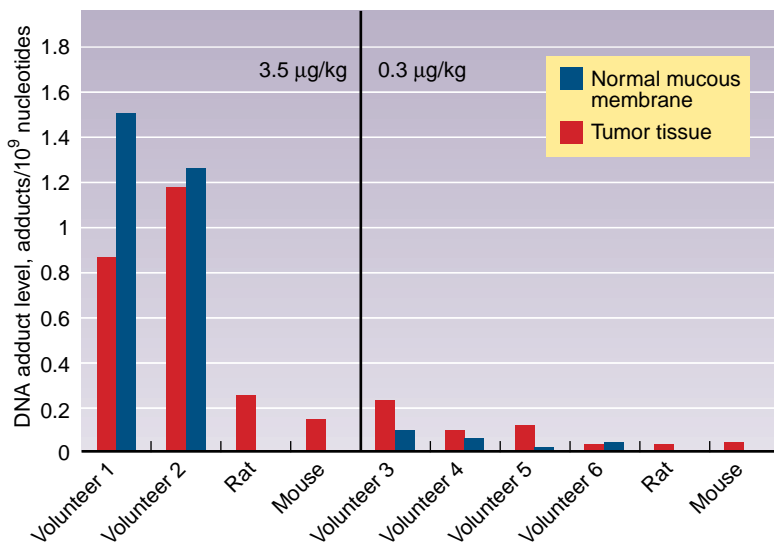
more sensitive to these substances than mice or rats. These results are important because researchers in the fields of toxicology, pharmacology, and nutrition currently make two basic assumptions: that data obtained from high-dose experiments can be accurately extrapolated to more typical environmental levels and that animal models are valid. While all the data are not yet in on the veracity of either assumption, AMS provides the sensitivity and precision needed to address each of them thoroughly.

Dingley performed the first AMS experiments using biomolecules labeled with tritium (hydrogen-3). With tritium-labeled PhIP and carbon-14-labeled MeIQx, she found comparable sensitivities at low doses. (The lowest doses were equivalent to the amount of PhIP you would take in when eating a single well-cooked hamburger.) These experiments demonstrated the feasibility of using tritium AMS for biomedical studies as well as the feasibility of using both tracers in experiments to study how two substances, given at once, may affect each other.

Tritium is widely used in biological tracing and has some advantages over carbon-14. It is relatively easy to label a molecule with tritium, whereas carbon-14-labeled molecules must be custom synthesized, which can be expensive. While the use of detection techniques other than AMS requires relatively high tritium dosages and large samples, AMS eliminates those disadvantages. In fact, Livermore's experiments have demonstrated that AMS could be used to detect tritium in biological samples with a 100- to 1,000-fold improvement over scintillation and other decay counting techniques.

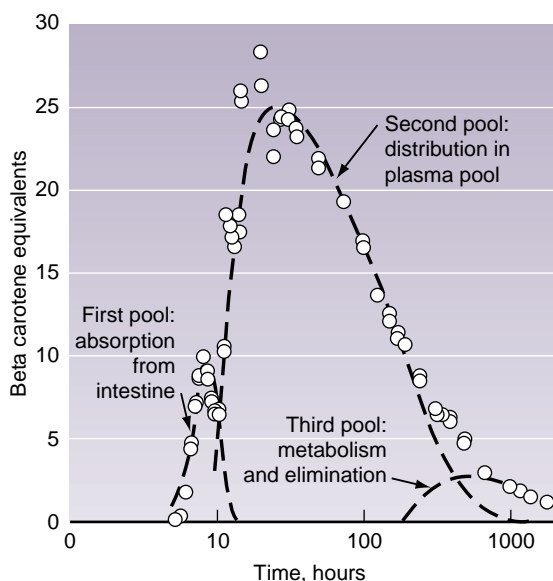
From Ants to Elephants

AMS is ideal for measuring extremely small samples, certainly the case when ants are the subject of study.



Rodents and humans exhibit significantly different responses to high and low doses of MeIQx, a chemical that appears in meat after it is cooked. MeIQx causes damage to DNA known as adducts.

Three "pools" of carbon-14-tagged beta carotene can be identified from blood, urine, and feces collected for 3 months after ingestion. The first peak is due to carotene metabolizing to retinol in the intestine, from where it is quickly excreted. The largest pool in plasma reaches its highest point a day after dosing but is then drawn into a deeper pool, perhaps the muscular tissue, over the next 5 to 10 days. This pool is slowly metabolized back to a circulating form and finally, in the third pool, is eliminated.



Research Opportunities Grow with New and Better Equipment

A continuing thrust of the Center for Accelerator Mass Spectrometry (CAMS) at Livermore has been the development and application of new AMS technologies as well as the improvement of existing ones. Three entirely new systems for measuring carbon-14, tritium (hydrogen-3), and plutonium-239 have come on line in recent weeks and months. Tweaking of the 15-meter-long, multipurpose AMS unit, in place since the inception of CAMS in 1989, never stops. After 11 years of development, the unit's cesium ion source is the most powerful in the world. Sample preparation methods and experimental protocols evolve and are standardized as Livermore's capabilities grow and as the demands of the biological research community expand. The focus now is to robotize the sample preparation process as much as possible.

For several years, researchers at Livermore have been working to develop a smaller, less expensive spectrometer dedicated to carbon-14 biological research. Biological AMS experiments can be run more quickly than most other AMS applications such as radiocarbon dating. A dedicated bio research unit would free the larger machine for other AMS work.

Testing for the first such small unit began in June (see [photo on p. 12](#)). One-tenth the size of the larger machine, the small spectrometer will provide higher throughput for biological samples. It also will serve as a testbed for new sample preparation and delivery technologies.

John Knezovich, director of CAMS, says, "These smaller units are just beginning to appear. Livermore is one of four institutions in the world using one. Now, virtually every AMS unit, of whatever size, is custom-built. But when they have been developed to the point where the cost for one is below \$1 million, then we can expect to see a proliferation of small AMS machines operating in universities and pharmaceutical companies, making significant scientific advances."



Livermore's Center for Accelerator Mass Spectrometry has developed specialized spectrometers for tritium (photo at left), heavy isotopes such as plutonium (photo at right), and carbon-14 (see p. 12), in addition to its large, multipurpose machine.

Livermore's new tritium system (below left) is the result of a collaboration with AccSys Technology of Pleasanton, California, and was funded by a National Institutes of Health/National Cancer Institute Small Business Innovation Research grant. The system was specifically designed to be small, simple, and inexpensive, making the technique of tritium AMS more broadly accessible.

Physicist Mark Roberts was primarily responsible for the system's design and for bringing it on line. Biochemist Karen Dingley, who performed the first AMS experiments using tritium as a biological tracer, has been developing experimental methods, including sample preparation and handling protocols. One of the system's first uses is in experiments to improve current methods to measure the rate at which cells divide. Cancer is a disease in which cells divide uncontrollably. Researchers want to know if exposure to cancer-causing chemicals results in an increased rate of cell division before a tumor appears. Normally, experiments such as this would require large doses of toxic chemicals or radioactive isotopes. With AMS, only a very small amount of tritium in drinking water does the trick. Then, the tritium in newly synthesized DNA can be measured. The level of tritium incorporation in DNA indicates the approximate rate at which new cells are synthesized and thus the rate of cell division.

Physicist Jeff McAnich has led the development of a new heavy-isotope spectrometer (below right) that makes it easier to run plutonium samples on a routine basis. The initial focus of this work is the measurement of plutonium concentrations and isotope ratios in urine bioassays of Marshall Islanders who are being resettled to Bikini Atoll and other islands used in the 1950s for weapons testing. The unit will be able to measure plutonium-239/240 as well as plutonium-241. Other heavy isotopes such as neptunium-237 may also be studied in the future.



CAMS worked with UC Riverside to follow the path of carbon-14-tagged food and insecticides through colonies of Argentine ants housed at the university. Once native to Brazil and Argentina, the Argentine ant is now the most prevalent pest around homes in California, the Caribbean, the Mediterranean, and South Africa. No available control strategy works effectively because each colony is home to several queens, any of which can regenerate the colony if other queens are destroyed. Learning how

nutrients make their way to the queens is essential to finding an effective control method.

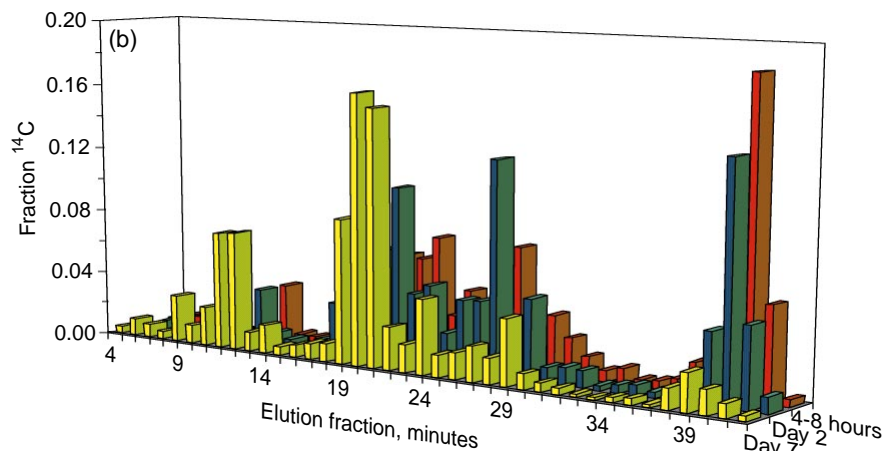
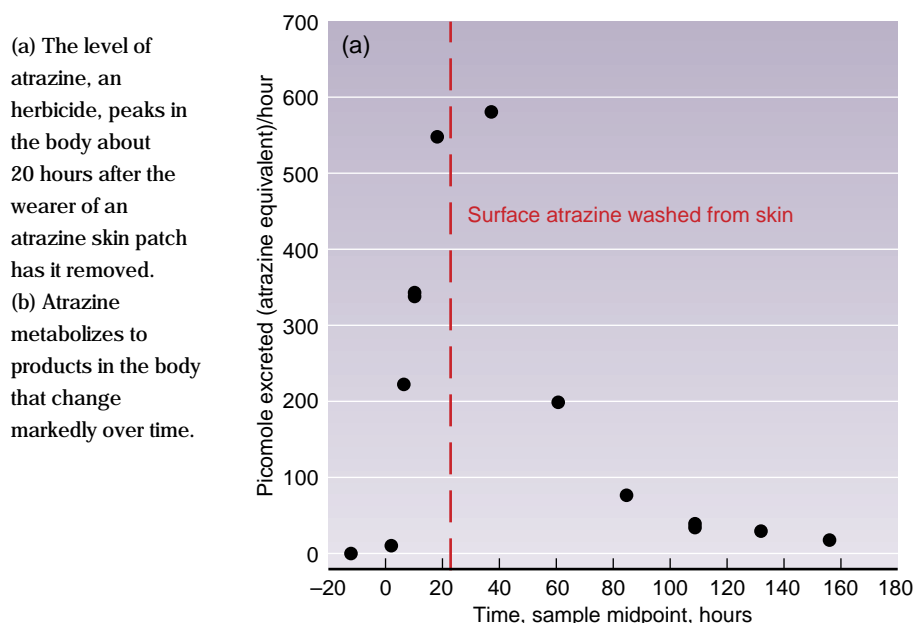
Work on nutrient dynamics was part of the largest AMS collaboration in place before the new NIH Research Resource was established. It involves CAMS and four UC campuses. Several campus-lab collaborations are ongoing at Livermore and other laboratories managed by UC, and the UC Office of the President has described this one as the most successful of all. Livermore's principal investigator for the

Consortium for Ultra-Low-Level Tracing is physicist John Vogel, assisted by Bruce Buchholz, who was trained as a nuclear engineer.

Vogel has been involved with CAMS from the start and has been responsible for many of the technological advances that have helped to establish AMS as an increasingly routine biomedical research tool. His work with UC Davis on nutritional studies has earned him the rank of adjunct full professor of nutrition. "One never knows where a career will go," he chuckles.

The CAMS-UC Davis experiment that followed a single dose of folic acid for 200 days was no fluke. Many over-the-counter products that we take with barely a thought stay in our bodies for a remarkably long time. A similar experiment followed a single dose of beta carotene in blood, urine, and feces samples for 3 months. The three primary "pools" of beta carotene or retinol, the vitamin A that beta carotene metabolizes to, are clearly visible, first in the intestine, then in blood plasma, and finally in a circulating form with a 38-day half-life. The existence and equivalence of the slowly changing pool that starts 9 days after dosing would be undetectable without AMS.

Metabolic studies of vitamins and other nutrients are of more than passing academic interest. At present, little is known about vitamin metabolism except that it seems to vary widely among individuals. Folic acid, one of the B vitamins, is required for the production of red blood cells, DNA, and RNA. A deficiency can cause anemia and is related to heart disease and certain birth defects. A deficiency in beta carotene can cause blindness, a serious problem in less developed parts of the world. In fact, a new variety of carrot-orange "golden" rice, rich in beta carotene, has recently been developed in Switzerland for use in undeveloped countries.



In 1998, a University of California at Riverside doctoral candidate won the annual award given by the *International Journal of Mass Spectrometry and Ion Processes* for best student paper. Her fundamental work in protein chemistry netted her a trip to Amsterdam as well as a cash prize. She could not have won without Livermore's biological AMS facilities because her subject, nematodes, supplied only the smallest of samples.

Dr. Andy Clifford, professor of nutrition at UC Davis, has headed all of this important nutrient metabolism work with CAMS. As a result of his research to date, he has been awarded follow-on grants from NIH to conduct definitive AMS studies in humans. His experiments are now under way using healthy human volunteers aged 18 to 60.

CAMS researchers are working with clinicians at UC San Diego to develop a diagnostic technique sensitive enough to detect the growth protein that cancerous tumors produce. Most of this protein stays at the site of the tumor, but a very small amount leaks out into the blood. Different kinds of tumors—ovarian, prostate, breast, liver, and so on—produce slightly different kinds of growth proteins. A blood test that could detect the protein would be an effective tool for an early diagnosis. The test could also be used to determine whether follow-up treatment was working; ideally, the protein level would drop to zero over time.

Yet another current study is of very large animals—elephants—but small samples. A hormone imbalance in bull elephants can produce raging behavior known as musth. The only way to obtain a sample from an enraged

elephant is from its feces. But most of that is undigested fiber, leaving researchers with little usable material from which to measure the carbon-14-tagged hormone precursors that the elephant has ingested. AMS comes to the rescue again.

Science in Revolution

For all this work to date, biological AMS is still very new. Knezovich acknowledges this, saying, "We at Livermore are still doing lots of missionary work, educating the research community about this powerful technology. But the NIH grant makes clear that the biological research community is convinced of the worth of AMS."

Livermore's AMS experts are part of an apparent revolution in which biology may be replacing physics as the haute discipline. Research in biosciences now accounts for more than 40 percent of federal funding for basic research, fueled in part by an aging population and increased needs for health care.

As the use of AMS for biological research grows and matures, related applications will quickly be found. CAMS has already participated in a study of the effects of atrazine, a

commonly used herbicide, on a group of volunteers in California who wore a skin patch of atrazine for 24 hours. Radioactivity in their urine was too low to be counted efficiently with liquid scintillation counting. But with AMS, uptake and elimination could be followed easily. Samples were even chromatographically separated to determine how the subjects metabolized the compound during exposure, a day after exposure, and several days after exposure. These biomarkers provide the data needed to develop an assay for occupational exposure to this chemical.

From agriculture to nutrition, from toxicology to chemotherapy—the potential uses of AMS for our better health are almost endless.

—Katie Walter

Key Words: biomedical research, carbon-14, Center for Accelerator Mass Spectrometry (CAMS), Marshall Islands, MeIQx, National Institutes of Health (NIH), nutrition, pesticides, PhIP, plutonium, radioisotope tracing, tritium.

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About the Scientist



JOHN KNEZOVICH has been the director of the Center for Accelerator Mass Spectrometry since May 1998. He received his B.A. in biology from the University of the Pacific in 1977 and his Ph.D. in chemical ecology from the University of California at Davis in 1983. He was previously a group leader for Environmental Chemistry and Toxicology. Knezovich is an environmental chemist with extensive experience in the design and application of experimental approaches for determining the fate, transport, and toxicity of contaminants in the environment. He originally came to Lawrence Livermore as a student guest in 1977 and returned as a postdoctoral scientist in 1983. Knezovich serves on University of California, state, and federal advisory panels that oversee research on toxic substances. He has written more than 60 scholarly articles and publications.

Mighty Small Dots

... nanoscience and nanotechnology will change the nature of almost every human-made object in the next century.

—The Interagency Working Group on Nanotechnology, January 1999

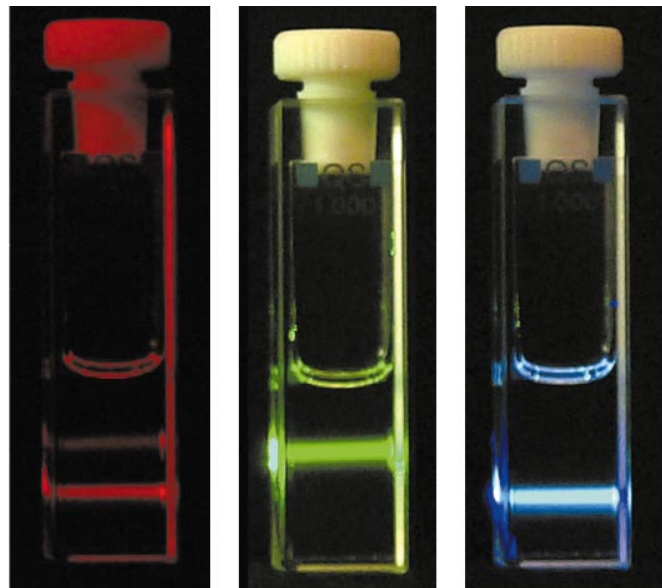
SMALLER . . . smaller . . . smaller. In the semiconductor industry, this mantra translates to faster . . . faster . . . faster. The question is, how small can you go?

At Lawrence Livermore National Laboratory, the answer may be: as small as quantum dots. Physicist Howard Lee and his team of Laboratory and University of California at Davis researchers have been exploring these entities, which are about a single nanometer (a billionth of a meter) in size and made out of material such as silicon. Lee explains, "Imagine taking a wafer of silicon and cutting it in half again and again and again, until you have a piece containing about a hundred to a thousand atoms. That's the size we're looking at."

The small size results in new quantum phenomena that yield some extraordinary bonuses. Material properties change dramatically because quantum effects arise from the confinement of electrons and "holes" in the material (a hole is the absence of an electron; the hole behaves as though it were a positively charged particle). Size changes other material properties such as the electrical and nonlinear optical properties of a material, making them very different from those of the material's bulk form. If a dot is excited, the smaller the dot, the higher the energy and intensity of its emitted light. Hence, these very small, semiconducting quantum dots are gateways to an enormous array of possible applications and new technologies.

"For years," says Lee, "scientists have been trying to make silicon emit light efficiently and in the visible range. This has been one of the holy grails of science." In 1990, researchers from Europe made porous silicon emit red light and attributed its color to quantum confinement arising from the small size. Since then, many other research institutions and commercial companies have taken an interest in quantum dots and have made silicon dots that emit at frequencies higher in the spectrum, in the much-sought-after green and blue ranges. In general, these higher energy emissions tend to be difficult to reproduce and not well understood.

"Here at the Laboratory," says Lee, "we have made silicon and germanium quantum dots that emit light throughout the visible spectrum—from the infrared to the ultraviolet. What makes our dots unique is that their luminescence can be tuned to any wavelength over a broad spectral range and be stable under ambient conditions. No one else has done this. We also believe we understand the underlying physics."



Howard Lee and his colleagues have synthesized silicon and germanium quantum dots ranging in size from 1 to 6 nanometers. The larger dots emit in the red end of the spectrum; the smallest dots emit blue or ultraviolet.

Smaller Is Beautiful

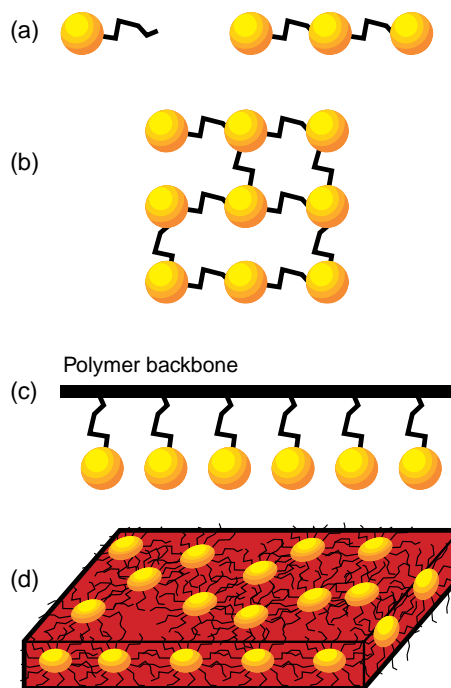
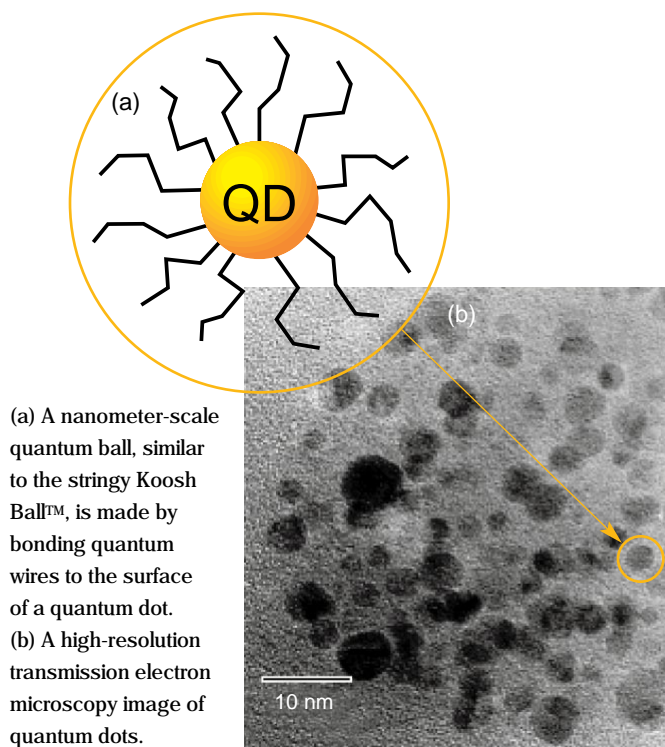
The color of the emitted light depends on the size of the dots: the larger the dot, the redder the light. As the dots shrink in size, the emitted light becomes shorter in wavelength, moving toward the blue. A rainbow of colors can be emitted from a single material simply by changing the dot size.

One possible use for this rainbow of colors is in biosensors used to detect agents of biological warfare. "Present-day fluorescence-based biosensors depend on organic dyes to tag the agents. The dyes luminesce to tell you whether any harmful bioagent is present," says Lee. "The problem is, dyes luminesce with a broad spectral width, which limits their effectiveness to a small number of colors. Furthermore, they degrade. Using quantum dots instead of dyes, the whole spectrum is available and there is little degradation over time."

"With all these different colors, it's now possible to make light-emitting diodes (LEDs) from quantum dots," says Lee. "We've come up with a process so easy you can almost do it in your garage. We can put these dots in a polymer and make thin films that are 1,000- to 2,000-angstroms thick. This means we can create precisely tuned blue or green LEDs."

Another future use for quantum-dot LEDs is to emit white light for uses in laptop computers or as internal lighting for buildings or cars. Lee and his team have discovered they can—by controlling the amount of blue in the emission—control the "flavor" or "tone" of the white light as well.

Quantum dots are also possible materials for making ultrafast, all-optical switches and logic gates. "We can make all-optical switches and logic gates that work faster than 15 terabits a second," says Lee. For comparison, the Ethernet



With molecular tethers to link them together, quantum dots become the building blocks of nanostructures. They can be linked together as (a) molecules, (b) lattices, (c) attached to a polymer backbone, or (d) incorporated into a polymer thin film.

generally can handle only 10 megabits per second. Quantum dots provide a remarkable millionfold improvement in speed. Other possible applications are all-optical demultiplexers (for separating various multiplexed signals in an optical fiber), all-optical computing, and encryption.

The frantic pace of innovation in semiconductors requires that integrated circuits be made with ever smaller features to carry more data and do it faster (see *S&TR*, March 1998, pp. 24–26, and November 1999, pp. 4–9). Most semiconductor fabrication processes start with a silicon wafer and etch away unneeded wafer material. Quantum dots, Lee explains, allow a bottom-up approach: “If you only need 100 atoms, then that’s what you make. And a single quantum dot can function as a microelectronic unit such as a transistor to form the basis of nanoelectronics.” At sizes of 1 to 6 nanometers, billions of dots will fit on the head of a pin. They will have incredibly fast operating speeds—1 picosecond or less—and extremely low power requirements.

Connecting the Dots

In a current Laboratory Directed Research and Development project, Lee is developing quantum wires to connect the dots together in a variety of configurations. As Lee notes, “If you use regular or even microscopic connections to link these dots, the size of the connections could destroy any useful quantum effects and defeat the purpose.”

The quantum wires are molecular tethers made of organic compounds chemically bonded to the surface of the dot. They can be of various lengths—the longest created to date is about 12 angstroms long—and serve multiple functions. They can be

electrically or optically active molecular structures. The wires on the dots add up to a nanometer-scale version of the popular kids’ toy, the stringy Koosh Ball™.

Using these wires and dots, Lee and his team are developing new nanostructures with quantum dots as the building blocks. The team is linking the dots in various one-, two- or three-dimensional configurations—as a molecule, a lattice, or attached to a polymer backbone. The molecular tethers act like electrical wires to the dots or as a way to control the interaction of connected dots.

Into the Wild, Blue Yonder

Quantum-dot LEDs, particularly those that provide the hard-to-reach blue end of the spectrum, appear to be key to opening any number of exciting technological advances in the fields of full-color, flat-panel displays; ultrahigh-density optical memories and data storage; backlighting; and chemical and biological sensing. “We have also explored the use of quantum dots for blue lasers,” notes Lee. “In 1999, we demonstrated that lasing may be possible with these quantum dots, opening the door to a new class of blue lasers that have intriguing applications for both the private sector and the missions of the Department of Energy.”

—Ann Parker

Key Words: all-optical switch, demultiplexer, light-emitting diode (LED), logic gate, quantum dot, quantum effects, semiconductor.

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Each month in this space we report on the patents issued to and/or the awards received by Laboratory employees. Our goal is to showcase the distinguished scientific and technical achievements of our employees as well as to indicate the scale and scope of the work done at the Laboratory.

Patents

Patent issued to	Patent title, number, and date of issue	Summary of disclosure
Glenn Allyn Meyer Dino R. Ciarlo Booth Richard Myers Hao-Lin Chen George Wakalopoulos	Rigid Thin Windows for Vacuum Applications U.S. Patent 6,002,202 December 14, 1999	A thin window that stands off atmospheric pressure is fabricated using photolithographic and wet chemical etching techniques and comprises at least two layers—an etch stop layer and a protective barrier layer. The window structure also comprises a series of support ribs running its width. The windows are typically made of boron-doped silicon and silicon nitride and are useful in instruments such as electron beam guns and x-ray detectors. In an electron beam gun, the window does not impede the electrons and has demonstrated outstanding gun performance and survivability during the gun-tube manufacturing process.
William Peter Van Antwerp John Joseph Mastrototaro Stephen M. Lane Joe H. Satcher, Jr. Christopher B. Darrow Thomas A. Peyser Jennifer Harder	Detection of Biological Molecules Using Boronate-based Chemical Amplification and Optical Sensors U.S. Patent 6,002,954 December 14, 1999	Method of determining the concentration of biological levels of polyhydroxylated compounds, particularly glucose. The method uses an amplification system that is an analyte transducer immobilized in a polymeric matrix. The system can be implanted under the skin and is biocompatible. When exposed to an optical source, the amplification system produces a signal capable of being detected outside the skin. Quantification of the analyte of interest is achieved by measuring the emitted signal.
M. Allen Northrup Conrad M. Yu Norman F. Raley	Process for Forming a Porous Silicon Member in a Crystalline Silicon Member U.S. Patent 6,004,450 December 21, 1999	Fabrication and use of porous silicon structures to increase the surface area of heated reaction chambers, electrophoresis devices, thermopneumatic sensor actuators, chemical preconcentrators, and filtering or control flow devices. In particular, such high-surface-area or specific-pore-size porous silicon structures will be useful in significantly augmenting the adsorption, vaporization, desorption, condensation, and flow of liquids and gases in applications that use such processes on a miniature scale. Devices that will benefit from a high-surface-area, porous-silicon structure include sample preconcentrators designed to absorb and subsequently desorb specific chemical species from a sample background; chemical reaction chambers with enhanced surface reaction rates; and sensor-actuator chamber devices with increased pressure for thermopneumatic actuation of integrated membranes. Devices that benefit from specific pore-size porous silicon are chemical-biological filters and thermally activated flow devices with active or adjacent surfaces such as electrodes or heaters.
Lawrence W. Hrubesh John F. Poco Paul R. Coronado	Method for Producing Hydrophobic Aerogels U.S. Patent 6,005,012 December 21, 1999	A method for treating a dried monolithic aerogel containing nondispersed particles with an organometallic surface-modifying agent to produce hydrophobic aerogels. The dried, porous hydrophobic aerogels contain a protective layer of alkyl groups, such as methyl groups, on the modified surfaces of the aerogel pores. The alkyl groups at the aerogel surface typically contain at least one carbon-metal bond per group.
John F. Holzrichter	Methods and Apparatus for Nonacoustic Speech Characterization and Recognition U.S. Patent 6,006,175 December 21, 1999	A simultaneous recording of electromagnetic wave reflections and acoustic speech information is used to define the positions and velocities of the speech organs during the articulation of each acoustic speech unit. Well-defined time frames and feature vectors can be formed to describe the speech. The feature vectors can uniquely characterize the speech unit being articulated during each time frame. The onset of speech, rejection of external noise, vocalized pitch periods, articulator conditions, accurate timing, identification of the speaker, acoustic speech-unit recognition, and organ mechanical parameters can be determined.

Patent issued to	Patent title, number, and date of issue	Summary of disclosure
Alan F. Jankowski Daniel M. Makowiecki Glenn D. Rambach Erik Randich	Hybrid Deposition of Thin-Film Solid-Oxide Fuel Cells and Electrolyzers U.S. Patent 6,007,683 December 28, 1999	The use of vapor deposition techniques enables synthesis of the basic components of a solid-oxide fuel cell, namely, the electrolyte layer, the two electrodes, and the electrolyte–electrode interfaces. The vapor deposition techniques are used in the three critical steps of material synthesis to produce a thin-film solid-oxide fuel cell. The electrolyte is formed by reactive deposition of essentially any ion-conducting oxide, such as defect-free, yttria-stabilized zirconia by planar magnetron sputtering. The electrodes are formed from ceramic powders sputter-coated with an appropriate metal and sintered to a porous compact. The electrolyte–electrode interface is formed by chemical vapor deposition of zirconia compounds onto the porous electrodes to provide a dense, smooth surface on which to continue the growth of the defect-free electrolyte, whereby a single fuel cell or multiple cells may be fabricated.
Stephen P. Vernon Natale M. Ceglio	Maskless Deposition Technique for the Physical Vapor Deposition of Thin-Film and Multilayer Coatings with Subnanometer Precision and Accuracy U.S. Patent 6,010,600 January 4, 2000	A method for the production of axially symmetric, graded- and ungraded-thickness thin-film and multilayer coatings that avoids the use of apertures or masks to tailor the deposition profile. A motional averaging scheme permits the deposition of uniform-thickness coatings independent of the substrate radius. Coating uniformity results from an exact cancellation of substrate-radius-dependent terms, which occurs when the substrate moves at constant velocity. If the substrate is allowed to accelerate over the source, arbitrary coating profiles can be generated through appropriate selection and control of the substrate center-of-mass equation of motion. Other distributions are obtained by direct mapping between the coating thickness and substrate equation of motion, which can be used to tailor the coating profile without the use of masks and apertures.
Paul B. Mirkarimi Claude Montcalm	Method to Adjust Multilayer Film Stress-induced Deformation of Optics U.S. Patent 6,011,646 January 4, 2000	A buffer layer located between a substrate and a multilayer to counteract stress in the multilayer and thus reduce or cancel out substrate deformation. The buffer layer, with a stress of sufficient magnitude and opposite in sign, provides a tunable, near-zero net stress in substrates such as an optic for an extreme ultraviolet lithography tool. Buffer layers have been deposited, for example, between molybdenum–silicon and molybdenum–beryllium multilayer films and their associated substrate, reducing the stress significantly, wherein the magnitude of the stress is less than 100 megapascals and near-normal incidence (5 degrees) reflectance of over 60 percent is obtained at 13.4 and 11.4 nanometers, respectively. The present invention is applicable to crystalline and noncrystalline materials and can be used at ambient temperatures.
Ward Small IV Peter Celliers	Single-Fiber Multicolor Pyrometry U.S. Patent 6,012,840 January 11, 2000	A fiber-based multicolor pyrometry setup for real-time, noncontact temperature and emissivity measurement. The system includes single optical fiber to collect radiation emitted by a target, a reflective rotating chopper to split the collected radiation into two or more paths while modulating the radiation for lock-in amplification (that is, phase-sensitive detection), at least two detectors (possibly of different spectral bandwidths, with or without filters to limit the wavelength regions detected), and optics to direct and focus the radiation onto the sensitive areas of the detectors. A computer algorithm is used to calculate the true temperature and emissivity of a target based on blackbody calibrations. The system components are enclosed in a light-tight housing, with provision for the fiber to extend outside to collect the radiation. The temperature range of the pyrometer system is determined by the spectral characteristics of the optical components.

Patent issued to	Patent title, number, and date of issue	Summary of disclosure
David R. Shafer	Reflective Optical Imaging System U.S. Patent 6,014,252 January 11, 2000	An optical system compatible with short-wavelength (extreme ultraviolet) radiation and comprising four reflective elements for projecting a mask image onto a substrate. The four optical elements are characterized in order from object to image as convex, concave, convex, and concave mirrors. The optical system is particularly suited for step-and-scan lithography methods. The invention increases the slit dimensions associated with ringfield scanning optics, improves wafer throughput, and allows higher semiconductor device density.
Gary E. Sommargren	Embedded Fiducials in Optical Surfaces U.S. Patent 6,014,264 January 11, 2000	Fiducials and a method for embedding them in optical surfaces are provided. Fiducials, or marks on a surface, are important for optical fabrication and alignment, particularly when individual optical elements are aspherical. Fiducials are used during the course of the polishing process to connect interferometric data and the equation describing the aspheric elements to physical points on the optic. By embedding fiducials below the surface and slightly outside the clear aperture of the optic, the fiducials are not removed by polishing, do not interfere with the polishing process, and do not affect the performance of the finished optic.

Awards

Jay Davis, former associate director of Earth and Environmental Sciences, was awarded the **Department of Defense Medal for Distinguished Public Service** for his work as director of the Defense Threat Reduction Agency (DTRA). Davis was recognized for his leadership in bringing together four separate organizations into an integrated, responsive defense agency to deter, reduce, and prepare for the threats of weapons of mass destruction.

DTRA also was recognized. It was awarded the **Joint Meritorious Unit Award** for its role in supporting the national nuclear deterrent force and implementing arms controls treaties. It has enhanced the capability of wartime commanders-in-chief to respond to chemical and biological attacks. Furthermore, it has eliminated a significant number of weapon systems threats in the former Soviet Union.

David M. Cooper, associate director for Computation and the Laboratory's Chief Information Officer, has been named by *Computerworld* as one of the **Premier 100 Information Technology Leaders for 2000**. Cooper is the only honoree from any government agency or national laboratory.

The selected 100 were those who most closely matched an information technology leadership index defined by the magazine's editors. Said editor-in-chief Maryfran Johnson, "*Computerworld* set out . . . to define who the most influential IT leaders are and what makes them tick. . . .The dramatic technological changes that Lawrence Livermore National Laboratory has made under

David Cooper's leadership exemplifies what *Computerworld* looks for in its 'Premier 100' honorees."

Cooper also was recently reappointed for a second term on President Clinton's Information Technology Advisory Committee. He had served on it since February 1997 as co-chair of the subcommittee on High Performance Computing and Communications.

The American Association of Engineering Societies (AAES) has elected **Ted Saito** as this year's chair. Saito served on the organization's board last year and previously sat as president of SPIE (International Society for Optical Engineering). He is a program leader in the Engineering Directorate.

AAES is the umbrella organization for more than 20 engineering societies. Saito's priority as chair will be to increase public awareness of the value that engineering brings to society. "Public surveys indicate most people feel they don't know enough about the profession," Saito says, "and they don't realize engineering's vital role in our economic well-being and national security." He will also be monitoring public policy issues and tracking legislation that would affect engineers, including a national portable retirement plan for them.

Saito has a B.S. in engineering and basic science from the U.S. Air Force Academy in Colorado, an M.S. in physics from the Massachusetts Institute of Technology, and a Ph.D. in physics from Pennsylvania State University.

Underground Explosions Are Music to Their Ears

A team of Livermore physicists and engineers is conducting explosive tests deep underground to provide important data for the nation's stockpile stewardship program. The experiments combine small amounts of chemical high explosives and weapons-grade plutonium to obtain new data about how plutonium and its alloys behave when strongly shocked and how that behavior changes as plutonium ages. The data are used to refine the nuclear simulation codes that run on supercomputers. The experiments, conducted at the Department of Energy's Nevada Test Site, are called subcritical because there is no nuclear detonation. Recently, in an effort to improve data collection and lower costs, the experiments have been conducted in expendable steel vessels that completely confine the experiments, making it unnecessary to carve out an underground alcove for each experiment.

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Biomedical Research Benefits from Counting Small

In just ten years, scientists at Livermore have taken accelerator mass spectrometry (AMS) a long way—from its first use in biomedical research to establishment of a National Institutes of Health National Research Resource for AMS at Livermore. The Center for Accelerator Mass Spectrometry at Livermore is unique in this country for its concentration on biological AMS; its work has been important in establishing AMS as a routine biomedical research tool.

Livermore scientists have led the technological advances necessary to make AMS a more effective, dependable, and economic tool for the biomedical, pharmaceutical, chemical, and clinical communities. They have developed and recently brought on line new AMS spectrometers, each designed to measure a specific biological tracer: carbon-14, tritium, and heavy isotopes such as plutonium-239. Ongoing research using Livermore's AMS facilities centers on measuring damaged DNA, studying the metabolism of nutrients and chemical substances, and detecting cancer.

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Preventing Nuclear Proliferation



Lawrence Livermore scientists are working with colleagues in the former Soviet Union to prevent the transfer of nuclear materials and nuclear expertise to other nations or even terrorists.

Also in September

- Lawrence Livermore's gas gun reveals "shocking" results about materials at extremely high pressures.
- Advanced computational techniques find the gems of information in enormous databases.
- The fourth-generation light source at the Stanford Linear Accelerator Center will yield the brightest x rays ever to probe the atomic secrets of matter.



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