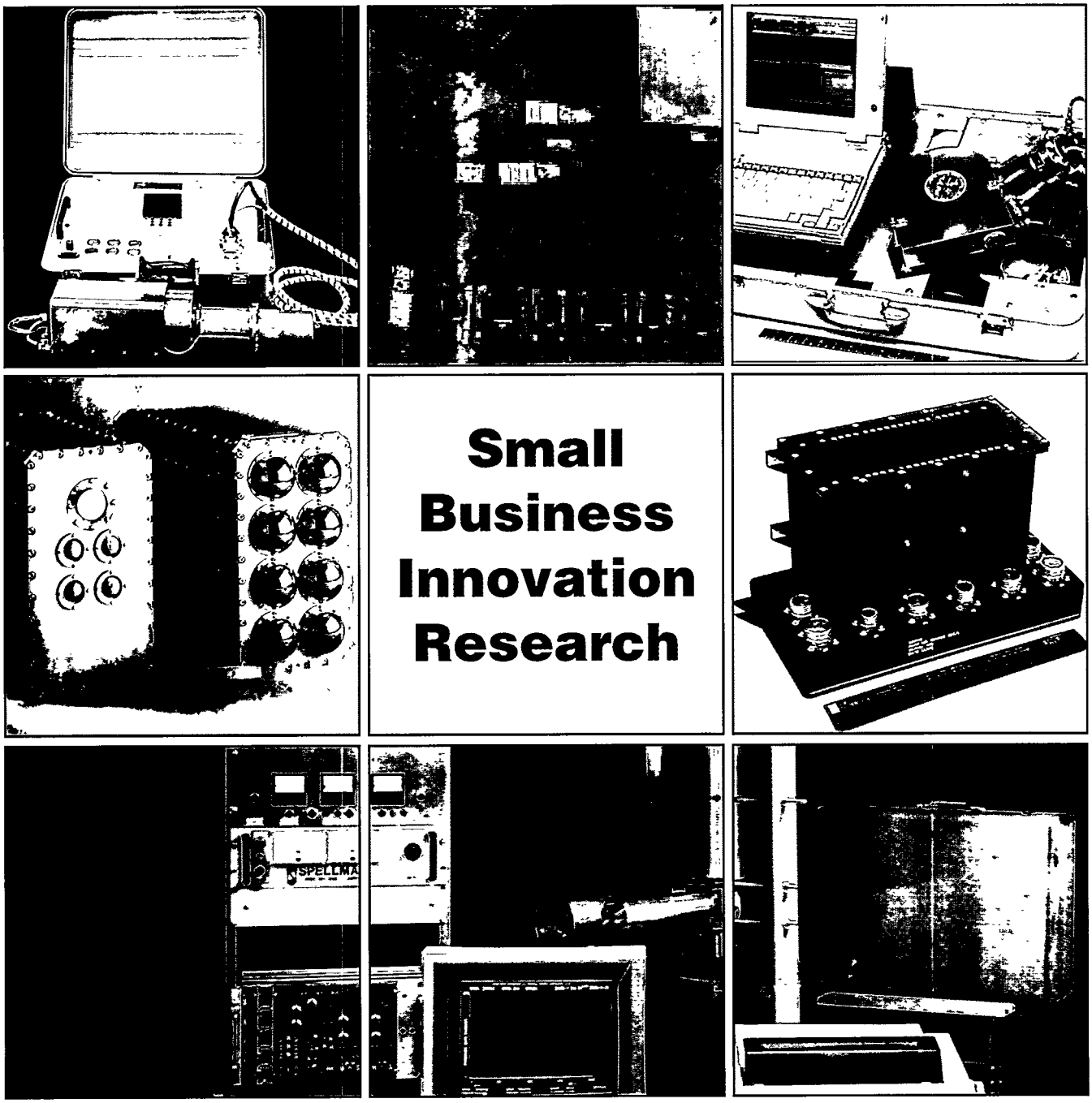
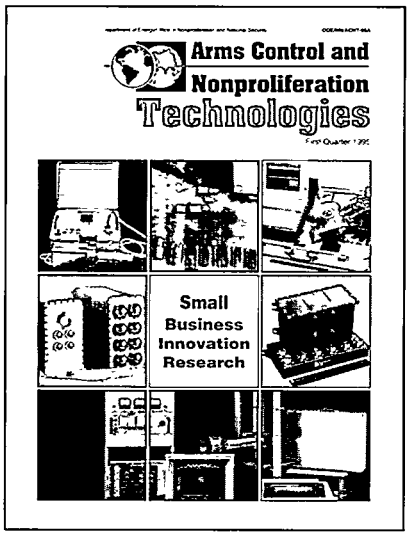
Arms Control and Nonproliferation Technologies

First Quarter 1995





About the cover

In this issue, we focus on SBIR—Small Business Innovation Research—the program managed by the DOE that stimulates the commercialization of technology. Federal funds help small businesses compete in developmental research. The photos ringing the center show SBIR and other technologies discussed in this issue.

The purpose of *Arms Control and Nonproliferation Technologies* is to enhance communication between the technologists in the DOE community who develop means to verify compliance with agreements and the policy makers who negotiate agreements.

Focus on SBIR

As our cover shows, the First Quarter issue for 1995 highlights the Small Business Innovation Research (SBIR) program. The SBIR program is managed by the DOE's Basic Energy Sciences program within the Office of Energy Research. Each year, the SBIR program solicits research ideas of interest to the DOE. Karl Veith, from the Office of Research and Development, explains the program in the first article, and three SBIR recipients present their particular projects.

An appreciation

This issue also marks a change in the general editors for *ACNT*. Leaving the publication for other assignments at LLNL are Sue Stull, the *ACNT* general editor since its first issue (as *Verification Technologies*) in 1989; and Cynthia Talaber, who joined the publication as a general editor in 1990. Replacing Sue and Cynthia are Gorgiana Alonzo and Penny Sanford, who are moving to *ACNT* from other editorial positions at LLNL. Also leaving *ACNT* is Peter Moulthrop, who has been the Scientific Editorial Consultant for several years.

Good luck, Sue, Cynthia, and Peter. We at DOE appreciate your fine contributions *to Arms Control and Nonproliferation Technologies.*

Michael F. O'Connell
DOE/*ACNT* Project Manager

Sue Stull *Cynthia Talaber*

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Focus on SBIR

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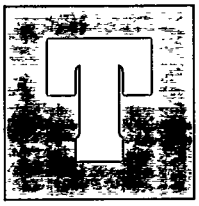


The Small Business Innovation Research Program supported by the Office of Nonproliferation and National Security

What is SBIR?

The Small Business Innovation Research (SBIR) program stimulates the commercialization of technology developed through federal research and development funds to help small businesses compete in the difficult area of developmental research. The SBIR program is managed by the Department of Energy's (DOE) Basic Energy Sciences program that is within the Office of Energy Research. Each fall, the DOE solicits grant applications covering many areas of interest to the DOE. The SBIR program conducts an annual competition for Phase I awards up to $75,000 for about six months to explore the feasibility of innovative ideas. Successful Phase I companies can apply for Phase II, the principal research and development effort, for awards up to $750,000 for a two-year period. In 1995, the DOE's SBIR program reached the $70-million level. While this is certainly a significant level of funding, it translates into only 200 Phase I and 80 Phase II awards per year. In 1994, the Phase I solicitation resulted in more than 2,000 proposals competing for the 200 awards. This makes the competition for the awards quite severe. A technical evaluation of a proposal requires three reviewers from three different institutions. This places a heavy workload on the SBIR's administrative office, the sponsoring office, and on the review process.

Focus on nonproliferation

The Office of Research and Development within the Office of Nonproliferation and National Security contributes more than $4 million to the SBIR program. During the last year, the Office took steps to ensure that the efforts of the awardees are well focused on the problems of nonproliferation. These steps encompass the full cycle of the SBIR program from the development of the solicitation statement to the commercialization of the product.

The topics for FY1994–95 were—

- Arms Control and Nonproliferation, Detection Technology
- International Atomic Energy Agency (IAEA) Special Safeguards.

The SBIR program was established in compliance with the Small Business Innovation Development Act of 1982 (Public Law 97-219). As prescribed in the legislation, the program is implemented in three phases. The program continues to strengthen the role that small innovation firms play in meeting the nation's research-and-development needs and in commercializing ideas developed through federally funded research and development.

The SBIR approach

The topic-development process was the first improvement. Each year, the SBIR program office requests research topics from the funding offices. The Office of Research and Development now actively spends time and effort soliciting suggestions for potential research areas, both from the other nonproliferation offices and from the national laboratories.

These suggestions are then reworked into related areas to provide the requisite topics and subtopics for a Phase I research solicitation publication.

The next step is the Phase I review and evaluation. Technical experts within the nonproliferation offices conduct preliminary screenings. Proposals that pass the preliminary screening are sent out for a full technical review. After the technical review, the final proposal rating is carefully developed by the Office of Research and Development, based on both merit and the potential contribution to nonproliferation.

Another improvement instituted by the Office of Research and Development is active interaction with the awardees. We are obligated to visit each Phase I contractor during the latter stages of the research project. During that visit, the facilities, employees, and capabilities are appraised to better assess the potential for success of the project if the company receives a Phase II award. In addition to this visit, the Office of Research and Development is trying to facilitate contact between the contractors and potential users for their systems. Arranging for the contractors to meet the users helps to acquaint the users with the research work and with company products that could fill existing needs of the users. This initiative, while it has seen limited application so far, shows signs of being of great value to both parties.

The last aspect of the Office's participation is continued contact with the small business into the Phase II development. We are suggesting that the Office of Nonproliferation and National Security/DOE personnel visit the site for a program review toward the end of the first and second years of the contract. This will enable us to monitor progress and to better integrate the results into the needs of the nonproliferation and security communities. The Office of Research and Development expects that these measures will help us use the SBIR program to its full potential.

As an example of the type of work being conducted under the SBIR program, we solicited the following articles, from Tamar Peli, Atlantic Aerospace Electronics Corporation, Waltham, Massachusetts, and from Kimberly Sharman, Telerobotics International Incorporated, Knoxville, Tennessee. Both companies have completed Phase I and have begun Phase II. The third article, from a team at the Lawrence Livermore National Laboratory, covers a topic that is in the process of joining the SBIR program. □

—Karl Veith, Office of Research and Development, DOE

Automated cueing to man-made objects via multi-spectral image

Figure 1 illustrates an overall system concept for the verification of arms control and nonproliferation. The concept resembles the Image Processing and Interpretation Centers idea first proposed by France at the 1978 U.N. Special Session on Disarmament to create an International Satellite Monitoring Agency (Final Document of the Tenth Special Session of the General Assembly). Our research focused on automating two functions in this concept: (1) detecting man-made objects, and (2) identifying violators by identifying objects and any changes associated with them.

Our Phase I SBIR program demonstrated the feasibility of an automated detection stage as part of an analyst station. The problem is to take the raw data coming into the station and reduce such data into a smaller number of sets, or cues, which can then be passed on to the next stage of processing—fractal-based discrimination.

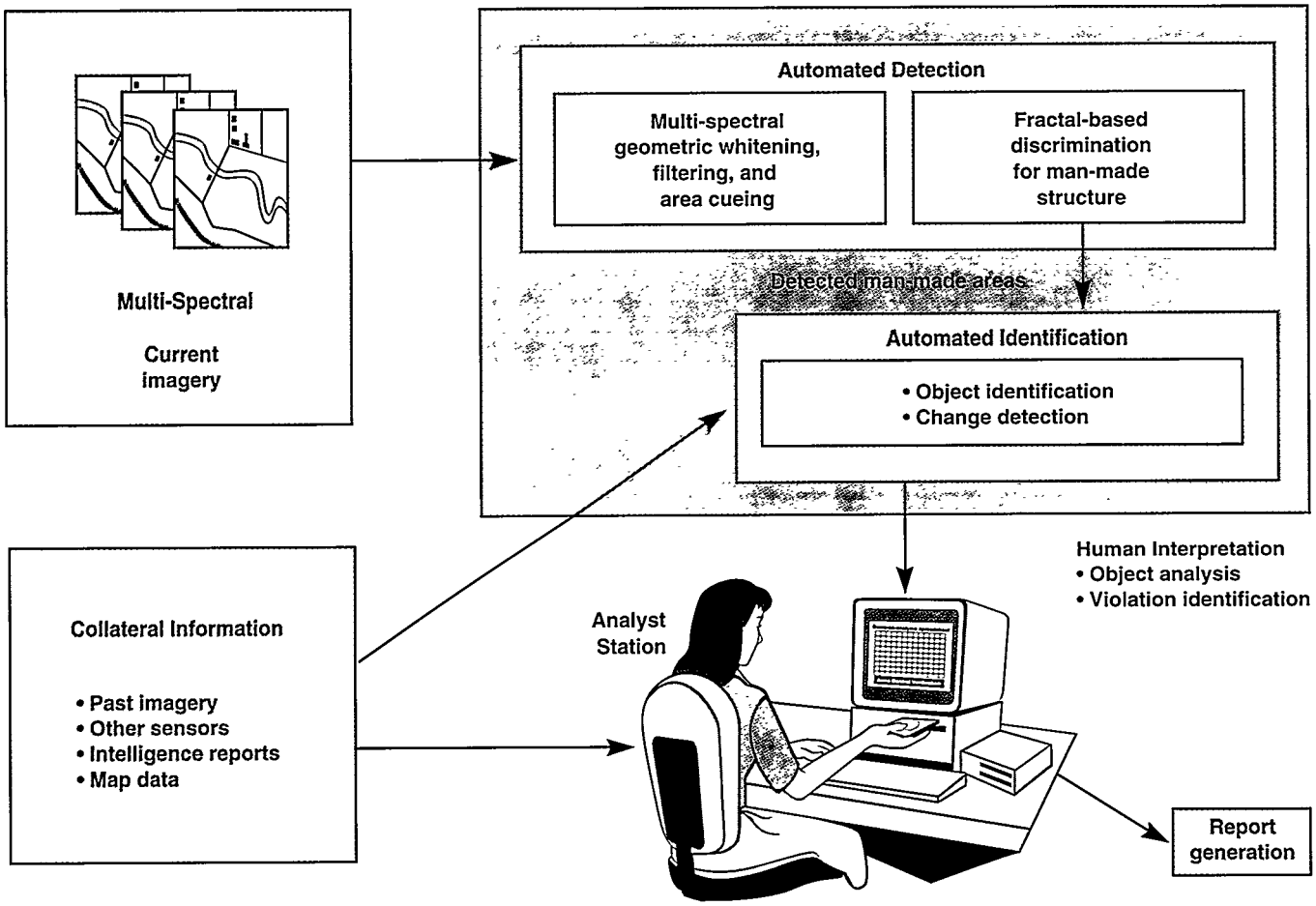
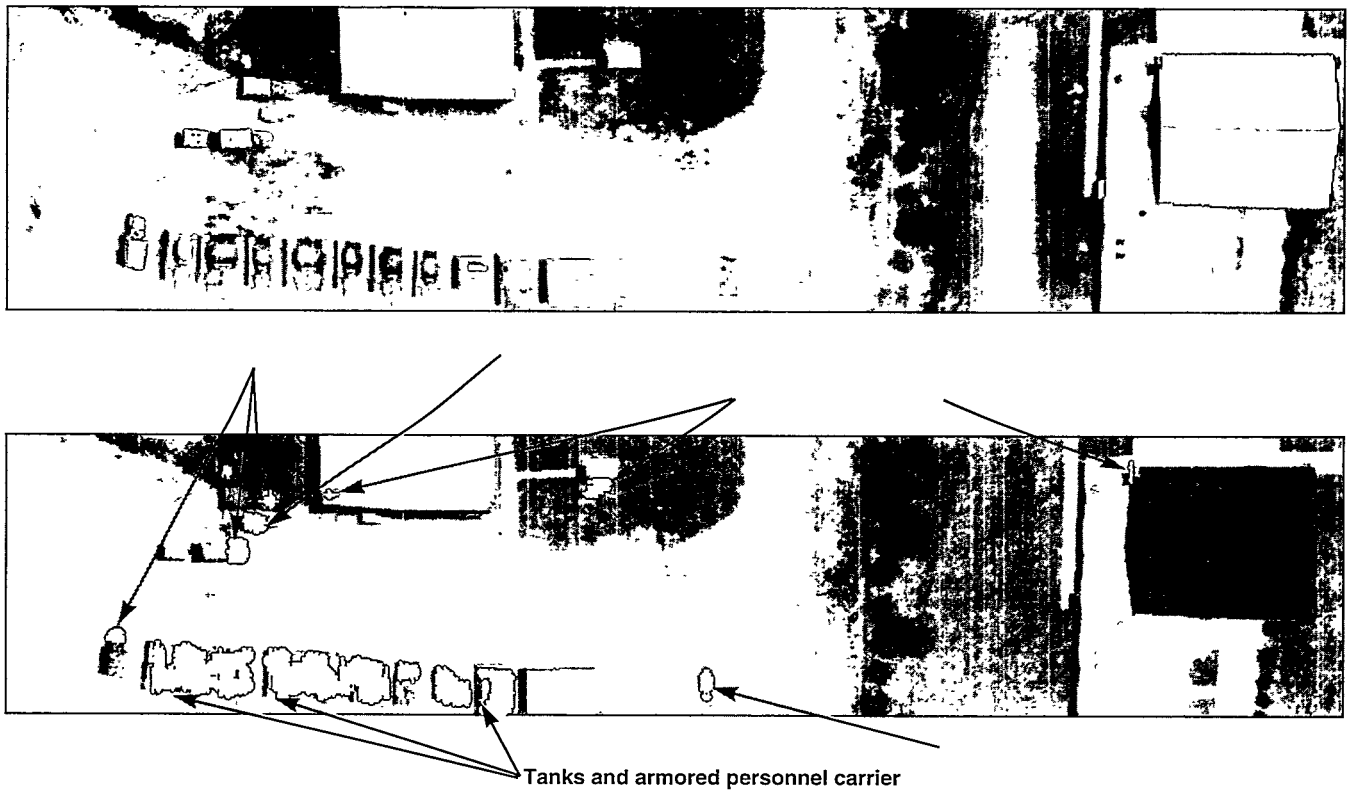


Figure 1. A system concept for the verification of arms control and nonproliferation.

Figure 2(a) shows a thermal infrared band of a sample multi-spectral image provided by EG&G. The image contains tanks, an armored personnel carrier, and truck cabs with camouflage paint, buildings, a fueling station, grass, and dirt. Figure 2(b) depicts the final set of cues to the camouflaged objects as fractal-based discrimination is applied. These appear as highlighted areas. All of the camouflaged vehicles were highlighted with few false cues.

The development of the fully automated process for detecting and identifying man-made activities (unconcealed and camouflaged) in multi-spectral imagery is currently continuing under a Phase II SBIR grant. The developed processes will be integrated into a commercial geographic-information-system software package and will provide automated screening for multi-spectral sensor data. □

—Contact: Tamar Peli, Atlantic Aerospace Electronics Corp., Waltham, Massachusetts, (617) 890-4200; Internet address is peli@aaec.com.



□ *Figure 2. (a) Thermal infrared band (b) fractal-based cues superimposed on near infrared band.*

Security systems get smart with advanced processing and thermal imaging

Two important ways to evaluate intrusion-detection systems are in their abilities to detect an intruder and the number of times they issue false alarms. The current state of technology in intrusion-detection systems is inadequate in these two categories. Perhaps the greatest shortcoming of present-day systems is their inability to distinguish between an actual intruder and a false stimulus. These include such things as light-level changes, dust, precipitation, shadows, and animals. In many cases, false stimuli result in an unreasonably high number of false alarms. The DOE is actively seeking a solution to human-presence detection suitable for applications such as outdoor perimeter control and nuclear vault surveillance. TRI, Inc. has developed and demonstrated the feasibility of a concept for a human presence detector (HPD) that uses advanced processing of thermal images to vastly improve system reliability while virtually eliminating false alarms. Because the HPD uses thermal information, only warm-bodied objects can trigger an alarm. Moreover, the HPD uses location, size, motion detection, and classification techniques to discriminate between human and non-human sources of heat.

When compared with conventional security systems, the HPD represents two significant paradigm shifts. The first is the use of thermal video over standard video. Thermal imaging offers several advantages: it is applicable for nighttime and daytime operation, it is less sensitive to changes in light levels, and it simplifies the process of identifying the object of interest in an image. While current thermal imaging technology will be used to demonstrate the HPD system capability, a commercial implementation of HPD will use the more affordable, uncooled thermal cameras that will be widely available in a few years.

The second paradigm shift in HPD is the use of multiple decisions in the detection process. The HPD analyzes data from a pair of thermal cameras to answer the following questions:
(1) Has a hot object entered the field of view?
(2) Has motion occurred?
(3) Is the object classified as human?
(4) Where is the object located?
(5) Is the size of the object within the range for a human?
A weighted sum of the results is used as the decision criteria for an alarm.

The significance of the multiple-decision approach cannot be underestimated. No single evaluation process can positively determine human presence. For example, unlike aircraft, which can be identified by known thermal signatures, humans cannot be detected based upon an expected thermal signature. The temperature range for a human varies greatly with clothing. Environmental factors also change temperature signatures. For instance, the thermal profile of a human standing outside on a winter day is going to be much colder than it would be indoors. Furthermore, warm-blooded animals can appear similar in temperature to humans. Motion detection alone, as evidenced by the number of false alarms that characterize video motion detectors, is not a reliable indicator of human presence. And, while object-recognition techniques are useful, they cannot always classify objects that are too unfamiliar. In short, the analytical results when considered individually can be inconclusive, but when fused provide a reliable determination of human presence.

In addition to exhibiting high false-alarm rates, many security systems are unreliable because of inadequate coverage or the lack of automation in the detection process. Live video surveillance systems, for example, are prone to miss events because they rely on human operators. Operators miss events for a number of reasons: they may have several screens to monitor at once, other duties may divert

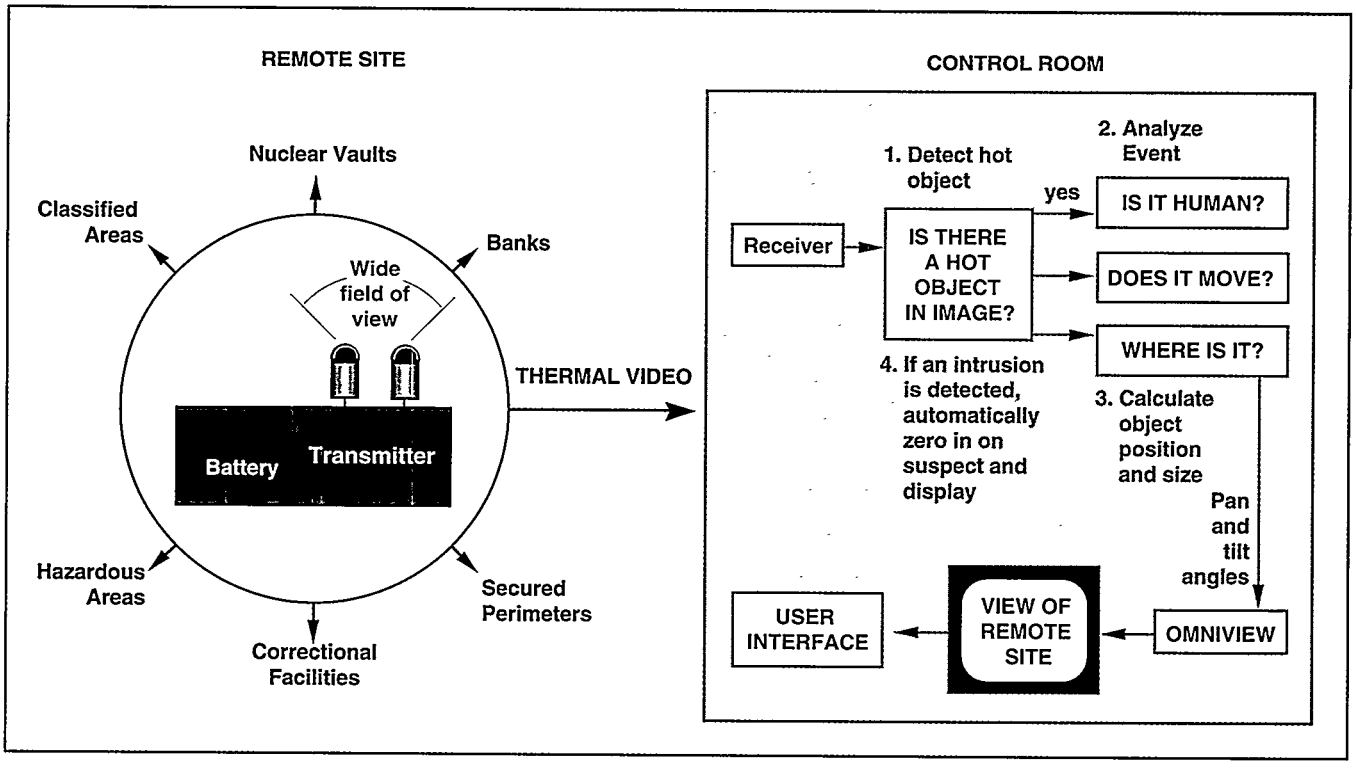
their attention, or a pan-and-tilt camera may be pointing in the wrong direction. The nature of surveillance (for the majority of the time nothing happens on the monitor) makes it difficult for operators to remain alert and attentive. The HPD provides the automatic detection required for high reliability.

System reliability is further increased with HPD's unique operator surveillance features. Omniview—a TRI-patented imaging technology that electronically pans, tilts, and zooms live video—provides the capability to automatically "zero in" on a suspect once a detection is made. The intruder can be tracked in a completely covert manner, since Omniview requires no moving parts that can be seen or heard. Furthermore, since Omniview removes the distortion associated with wide-angle optics, the HPD can offer large area coverage from a minimum number of cameras.

In a Phase I effort, TRI, Inc. developed a system concept for a human presence detector that is able to make a positive determination of a human with a very low false-alarm rate. The feasibility of the HPD concept was demonstrated through developing the automated detection process on a PC-based system and testing the system using thermal images. The next step in the HPD development will be a system prototype that responds in real time and can be trial tested at a DOE site.

The HPD system was designed to fill a need within the DOE for a reliable intrusion-detection system for nuclear vaults, secured perimeters, and other sensitive facilities. However, the HPD also has tremendous commercial value for use in museums, bank vaults, correctional facilities, and other high-security areas. The positive results attained thus far point to the viability of this approach for both military and industrial security applications. Given the existing market need for a reliable intrusion-detection system, the successful demonstration of HPD will provide the exposure necessary to take the product to market. □

—Contact: Kimberly Sharman, TRI, Inc., Knoxville, Tennessee, (615) 690-5600; Internet address: sharmank@omniview.com



□ *Figure 1. Human presence detection (HPD) system.*

A breakthrough in cooling system technology

Wide varieties of commercial equipment such as scientific instruments and detectors for industrial operations require very low, or *cryo*, temperatures to operate. Lawrence Livermore National Laboratory has designed a vibration-suppressed mechanical cooling system that efficiently provides low temperatures by reducing vibrational noise. Compared with traditional cooling technology, the new system is relatively inexpensive to produce and operate, and is environmentally friendly. Originally developed for a radiation detector, the new design could be adapted for many instruments that require cryo-temperatures and low vibration. The commercial potential for applications in refrigeration, air conditioning, and scientific instrumentation is unlimited.

LLNL was initially interested in a portable cooling system to use with a gamma-ray detector for measuring radioactive materials in the field. Routine field measurements have been both cumbersome and impractical because the detectors require very low-temperature liquid-nitrogen cooling. This traditional cooling technology adds weight to the detection system and requires a complicated support structure for proper use. Other cooling methods, like those in air conditioners, are not practical because of high power consumption, large amounts of mechanical vibration, and environmentally unfriendly coolants.

The new cooling technology is portable, requires low power, and uses environmentally inert helium (Fig. 1). LLNL's Isotope Sciences Division (ISD) supplied proof-of-principle funding. After the concept was demonstrated, the LLNL-ISD development team continued its work under the auspices of DOE's Office of Research and Development (NN-20). A patent application has been filed and LLNL is interested in licensing the technology.

Electro-mechanically cooled germanium detector

The application of this technology to electro-mechanically cool a germanium detector is shown in Fig. 1. To control vibration, which

□ *Figure 1. LLNL's electro-mechanically cooled germanium detector.*

markedly degrades detector performance, available hardware was modified to counter-balance the extraneous motions created by the cooler's motorized compressor. The LLNL-ISD design uses microprocessors to produce a counterforce to cancel undesirable movement. With less vibration, the compressor becomes more efficient, uses less energy, and lasts longer. Because the cooling system is portable and operates with significantly reduced vibrational noise, the germanium detector can easily be used in the field. The current prototype has operated for more than 4,500 hours, through multiple power cycles with an estimated life expectancy between 5 and 10 years.

Environmentally friendly

The LLNL-ISD cooling system uses small amounts of helium—a common, inert gas—as a coolant rather than the chlorofluorocarbons (CFCs) traditionally used in refrigeration systems and

The electro-mechanically cooled germanium detector provides—

Superior performance

High-resolution (low-energy germanium detector)
- 540 eV (0.4%) at 122 keV
- 430 eV (0.7%) at 59.4 keV

Durability
- Prototype cryo-cooler operated for more than 4,500 hours (more than 200 power cycles)
- Expected mean-time-to-failure (MTTF) of system is 5-10 years

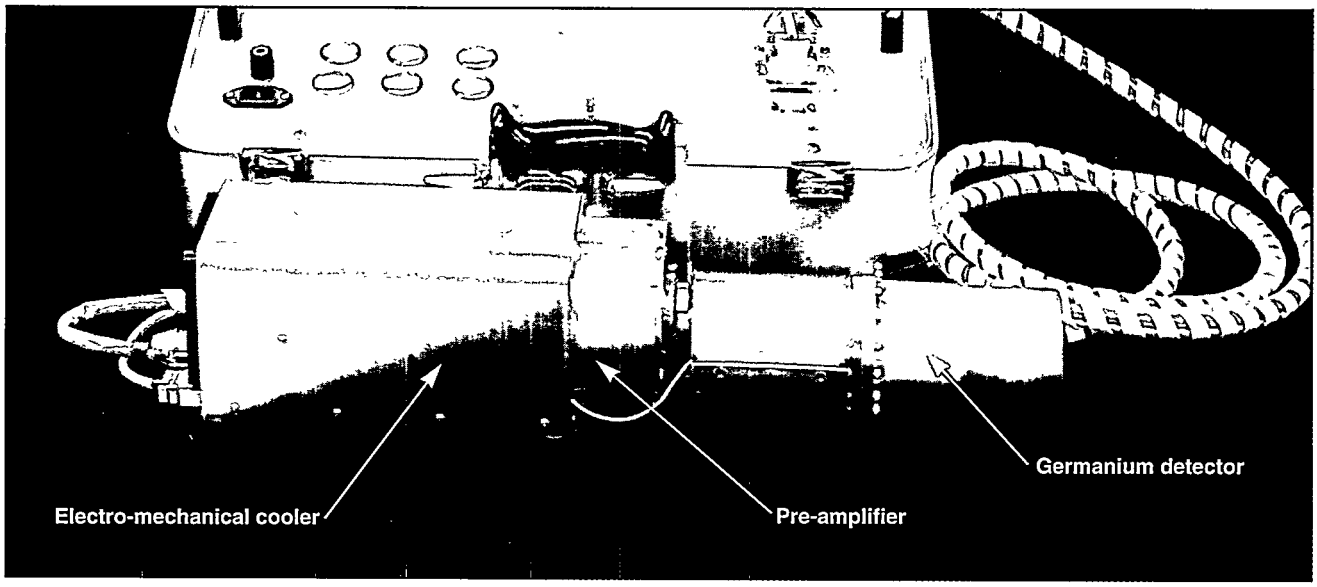
Temperature stability
- Cryo-cooler temperature controlled to ± 0.5 K at 78 K

Portability

Lightweight
- Less than 6.8 kg (detector/cryo-cooler)

No liquid nitrogen required
- Cooldown for low-emission germanium approximately 3–4 hours
- Cooldown for 50% high-purity germanium approximately 3–4 hours

Battery-operated
- 8–12 hours' operation with 11.4-kg Ni-Cd batteries
- Future development will include LANL's multi-channel analyzer (M^3CA) installed in the battery pack



which can harm the earth's ozone layer. Available CFC substitutes compromise any environmental benefit because their use requires larger and more energy-hungry compressors. The LLNL-ISD design not only uses benign helium, but the system's small size overcomes the compressor problem.

Retrofitting existing cryogenic systems

Another possible advantage of the new technology is the ability to retrofit existing detector systems that traditionally have used liquefied nitrogen for cooling. Liquefied nitrogen systems are bulky, labor-intensive, and require extensive safety systems. The LLNL-ISD design eliminates the potentially hazardous liquefied nitrogen and its attendant requirements. Existing systems such as those used for medical position emission tomography (PET) scans could be retrofitted with this new technology.

Benefits

The potential benefits of this new cooling technology are as wide-ranging as the applications. Highly sensitive gamma-ray measurements can easily be taken in the field without worrying about power consumption, weight, or vibrational interference. Because the cooling system was designed to work with commercial germanium detectors with minimal modifications, it is possible to retrofit existing liquid-nitrogen systems, thus eliminating liquid nitrogen refills and their attendant requirements. We believe that the cost savings alone from reducing the personnel, safety, and environmental monitoring expenses will easily pay for retrofitting in 12–24 months, depending on the operation. Other potential applications include refrigerators, air conditioning for cars, many types of scientific instruments, and solid-state electronics. □

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The APSTNG neutron probe

Ed Rhodes and Charles Dickerman
Argonne National Laboratory

An instrument called an associated-particle, sealed-tube, neutron generator (APSTNG) directs neutrons into unknown or hidden objects and determines their nuclear and chemical composition. Potentially detectable items include explosives, narcotics, toxic chemicals, nuclear weapons, and special nuclear materials (SNM).*

Transportable in a van, this probe could have treaty-verification, nonproliferation, and on-site inspection roles for preventing both commerce in and stockpiling of nuclear and chemical weapons of mass destruction and their construction materials. Another role could be to help establish effective export controls for the DOE Former Soviet Union Nonproliferation Export Control Project.

The APSTNG is a special small accelerator tube that interrogates the object of interest with a continuous, low-intensity cone of 14-MeV neutrons generated from the deuterium-tritium reaction. The alpha-particle associated with each neutron can then be detected (for a timing and direction reference). Gamma-ray spectra of resulting neutron reactions can identify fissionable materials and many elements (nuclides) having an atomic number larger than that of boron. Flight times determined from the detection times of the gamma rays and alpha particles can yield a coarse, three-dimensional (3D) position for each identified nuclide. Chemical substances can be identified by comparing relative spectral-line intensities with ratios of elements in reference compounds.

The source and emission detection systems can be located on the same side of the interrogated volume, allowing measurements when access is possible from only one side. The high-energy neutrons and gamma rays penetrate large objects and dense materials. Thus, this system allows examination of relatively thick and large sealed containers and areas behind walls, and also reduces deception caused by radiation shielding.

*This work was sponsored by the Department of Energy, Office of Nonproliferation and National Security (NN-20).

Principles of operation

The APSTNG neutron probe is diagrammed in Fig. 1. The object being interrogated might be baggage, cargo, or an item to be inspected under an arms-control treaty. Inside the APSTNG tube, deuterons are accelerated into a tritium target, producing 14-MeV neutrons isotopically. Each neutron is emitted at the same time an associated alpha particle travels in the opposite direction.

When a prompt (fast-neutron) reaction inside the cone produces a detected gamma ray, the time delay from the alpha pulse yields the position (depth) along the cone where the reaction occurred, because the geometry and the source neutron and gamma-ray speeds are known (5 cm/ns and 30 cm/ns, respectively). If a position-sensitive alpha detector is used, a coarse 3D image of reaction locations can be obtained from a single measurement orientation. Information for each event is digitized and stored in a personal computer (PC), along with flight time and gamma-ray energy. The PC controls measurements, calculates positions from the recorded data, and displays data, graphs, and images. Software can be developed for specific applications that will allow the PC to perform intelligent data analysis and interact with the operator to determine which items are sufficiently suspect to require further examination.

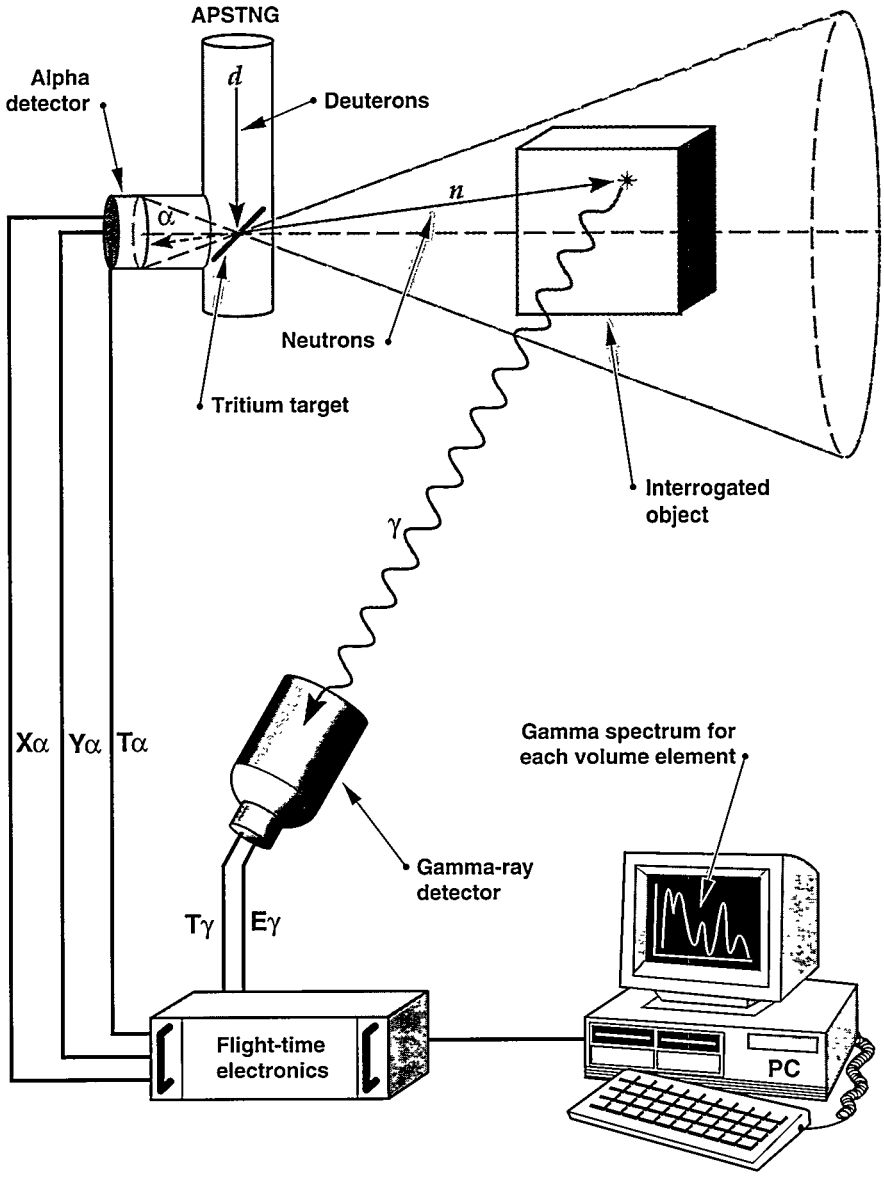
A model APSTNG system is shown in Fig. 2. In most applications, systems would probably include a specific array of

gamma-ray detectors, so as to maximize information obtained from each interacting neutron and minimize measurement time. In Fig. 3, a design concept depicts an array of 18 double-ended gamma-ray detectors around cargo being inspected for SNM.

Multiple detection modes

Fast-neutron inelastic scattering reactions in the object provide prompt gamma-ray spectra that can identify many nuclides. By choosing gamma lines of specific nuclides, a 3D position of each identifiable nuclide can be determined. By choosing appropriate nuclide intensity ratios, 3D positions of chemical compounds can be made.* The use of the time-correlated gamma-ray spectra, as discussed so far in relation to Fig. 1, is denoted the emissive gamma-ray imaging and spectroscopy (EGRIS) mode. Nearly all nuclides with atomic numbers above boron have distinctive gamma-ray spectra for the basic EGRIS mode, with reaction cross-sections of about 0.5 barn for 14-MeV neutrons.

For gamma rays above ~1 MeV, the background in the EGRIS mode is reduced because background counts can be accumulated only during the nanosecond-range correlation interval. Because gamma rays are emitted nearly isotopically in the neutron reactions, the neutron tube and gamma-ray detectors can be located on the same side of the interrogated volume, allowing measurements when access is possible on only one side. The EGRIS mode is the basic APSTNG detection mode, but it can be augmented by other detection modes for various applications. In all detection modes mentioned, no collimators for the source or detectors are used and little or no personnel radiation shielding



□ *Figure 1. Operational layout of the associated-particle, sealed-tube, neutron generator (APSTNG) system.*

*However, molecular bonds cannot be identified.

is needed, which improves detection efficiency and reduces system size and weight.

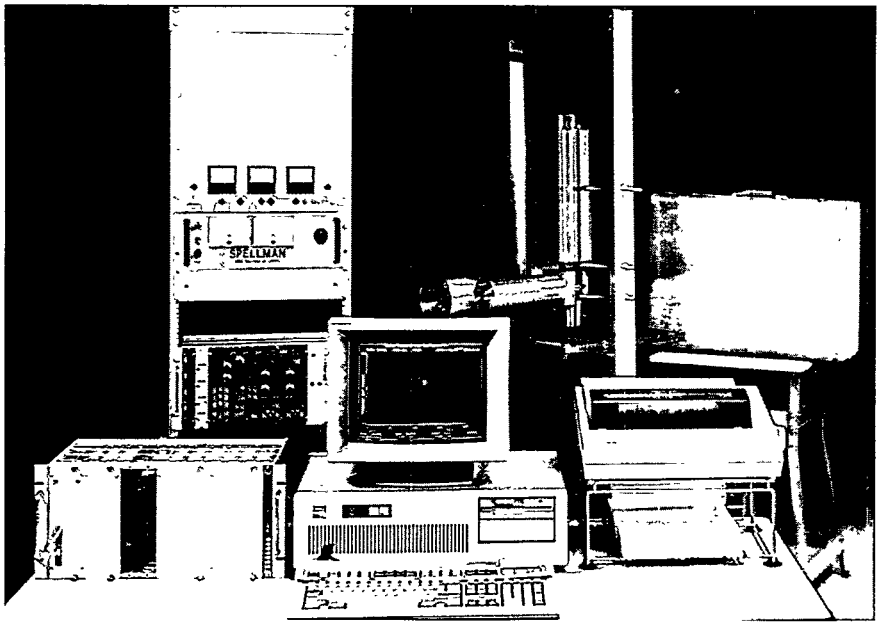
Other modes can collect complementary information. Gamma rays detected from thermal-neutron reactions can provide information on the presence of fissile materials or materials having a large capture cross-section for thermal neutrons. Also, if neutron detectors can be placed behind the object, the neutron beam in conjunction with the alpha detector (which determines the direction of the neutrons) can make a transmission image of the suspect object. In addition, the neutrons can be turned off to allow only gamma rays from the suspect device and its surroundings to be detected with the high sensitivity of the large gamma-ray detectors.

Treaty verification and nonproliferation applications

Potential treaty-verification applications include monitoring the output streams from dismantlement facilities and inspecting cruise missiles (declared to be conventionally armed) for hidden nuclear warheads. Nonproliferation roles might include monitoring SNM production facilities, monitoring International Atomic Energy Agency reactor facilities, monitoring ports of entry, inspecting radioactive waste for SNM, and conducting challenge inspections.

Fissionable materials are identified in the EGRIS mode by the characteristic induced-fission, gamma-ray spectrum (because their primary inelastic gamma-ray energies are well below 1 MeV), and the APSTNG system might be supplemented by neutron detectors that can identify the induced fission neutron spectrum (provided there is sufficient discrimination against gamma rays and scattered 14-MeV source neutrons). Other relevant materials would be identified by inelastic gamma rays induced by the neutrons. For example, Fig. 4 shows the integrated EGRIS gamma-ray energy spectrum for uranium blocks surrounded by an explosive simulant, with energy bands chosen to identify these materials. Spectra for volumes occupied by the uranium show only the fission band, and vice-versa.

Natural radioactivity from SNM that is not absorbed can be detected by turning off the neutrons. If it is desirable to further identify fissile SNM, a separate measurement can be performed in which moderating material is placed between the APSTNG tube and the interrogated object. It might be feasible then to identify particular fissionable nuclides by their capture gamma-ray spectra.

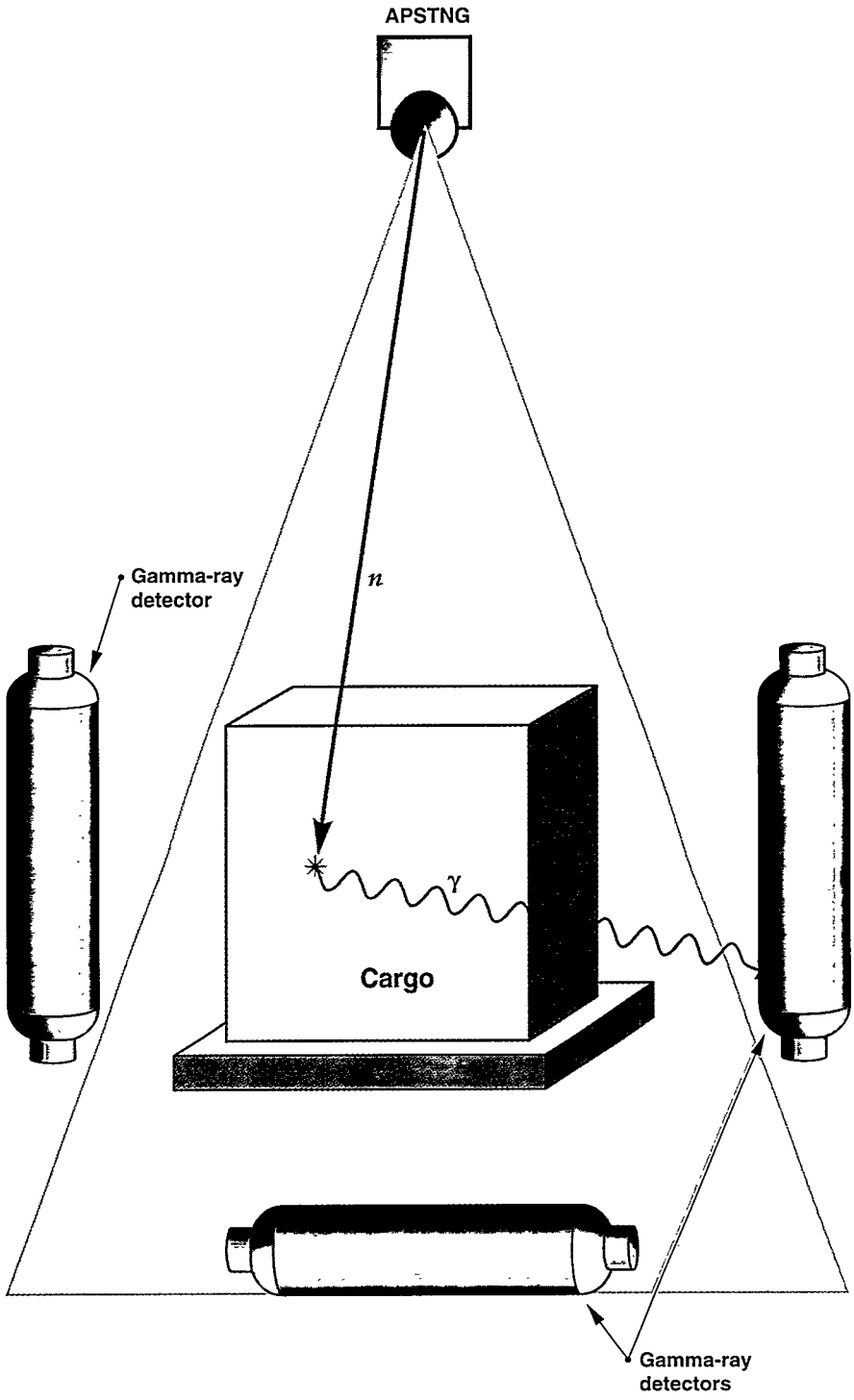


□ *Figure 2. Photo of an early-model APSTNG system. At the top right center of the photo is the APSTNG neutron accelerator tube. Proceeding counterclockwise and behind it is the larger NaI gamma-ray detector. At the top left is a rack containing the accelerator control system, high-voltage supply, and flight-time electronic modules. Below are the data-acquisition electronics and a personal computer with printer. At the top right is a briefcase that provides a sense of scale.*

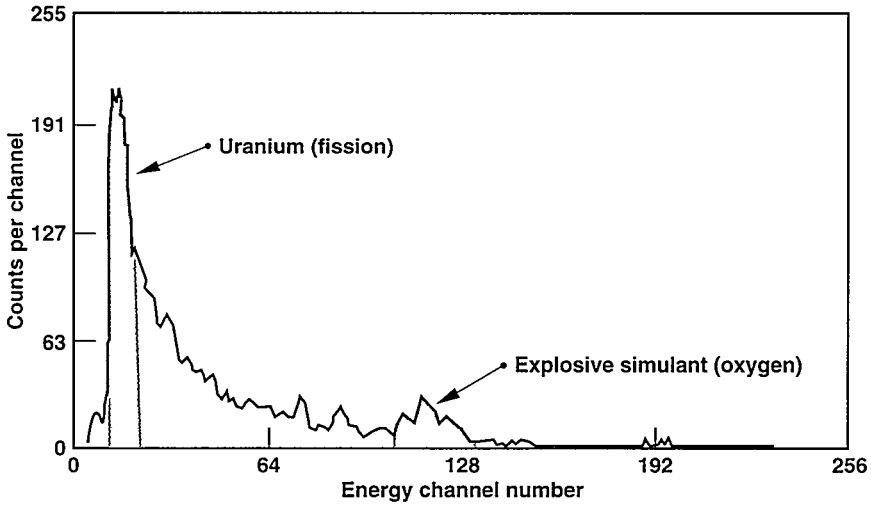
Hidden neutron shielding can be detected in cases where neutron detectors can be placed behind the interrogated object. Our system has significant built-in deception resistance against gamma-ray shielding for gamma rays above ~1 MeV because of their penetration, so an anomalously high loss in gamma-ray count rates would likely be observed.

In a nuclear-warhead-dismantlement application, both the SNM and non-SNM output streams from dismantling facilities could be monitored. The non-SNM stream could be checked for hidden SNM to an agreed-upon sensitivity level by using the EGRIS mode to check for the presence of uranium and plutonium, followed by a measurement with inspector-added moderator to enhance signals from plutonium or highly enriched uranium. The SNM stream could be checked remotely for coarse shapes and sizes and total amounts of SNM (the limitation on the use of the technology will be governed by classification guides). The ability of the high-energy APSTNG neutrons and induced gamma rays to penetrate moderating materials designed to stop fission neutrons could be of use in various applications, such as inspection for hidden nuclear warheads in cruise missiles declared to be conventionally armed and portal surveillance against uranium and plutonium smuggling.

For most of these applications, increased detectability will be more important than reduced



☐ *Figure 3. Emissive gamma-ray imaging and spectroscopy (EGRIS) APSTNG system proposed for use in detecting special nuclear material (SNM). There are 18 long NaI detectors, each with a photomultiplier on each end (5 detectors are hidden behind each of the 3 detectors shown).*



□ *Figure 4. Integrated EGRIS gamma-ray energy spectrum for uranium blocks surrounded by an explosive simulant.*

intrusiveness. Spatial resolution will be low compared with x-radiography norms, given the limitations in signal rate and time resolution. Energy resolution of the current reference NaI detectors will be low compared with high-resolution spectroscopy norms. However, if necessary, intrusion can be controlled by limiting measurement time, gamma-ray-detector energy resolution, system time resolution, and/or alpha-detector spatial resolution. □

Lithium-doped fullerene neutron detector

Fullerenes are a recently discovered class of materials that constitute a third stable form of pure carbon (along with graphite and diamond). These spherical closed carbon networks, of which the soccer-ball shaped C_{60} is the archetypical member, are named for their similarity to the geodesic domes of R. Buckminster Fuller. Current research into fullerenes is focused on their electrical properties, particularly on fullerenes doped with an electron donor such as potassium. Other current areas of research include the synthesis of doped fullerenes, with the dopant atoms located inside or outside the fullerene molecules; synthesis of fullerene molecules made up of an increasing larger number of carbon atoms, termed *giant fullerenes*; and the synthesis and properties of *nanotubes*, long tubes whose surface is a curved graphitic network.

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NCCOSC RDT&E DIV, 49590 Lassing Road, San Diego, CA 92152-6171

With today's increasing awareness of the need for control and identification of fissionable materials, a small, sensitive, robust neutron detector has more applications than ever. We are studying a new material—lithium-doped C_{60}—that holds promise for the development of a novel solid-state neutron detector. There are many potential advantages to such a solid-state detector. It would be insensitive to vibration and shock, would have a high neutron absorption cross-section, would not require high gas-pressure containment, would not be a scintillation device, and the presence of a large amount of carbon would act as a moderator for fast neutrons. Consequently, the detector would overcome many of the deficiencies of existing detectors. We have devised a new method of synthesizing doped C_{60} and are studying ways of using this new material for neutron detection under the sponsorship of DOE's Office of Nonproliferation and National Security.

Electronic properties of doped fullerenes

The recent discovery that pure carbon can form closed spherical structures, called fullerenes, has generated a flurry of research. Figure 1 shows the molecular structure of C_{60}. In the undoped state, the material is chemically stable, physically durable, and electrically nonconducting.

The electronic and magnetic properties of these fascinating materials have been under intense study since the discovery that, when doped properly with alkali metals, the compounds are superconducting.[1] Among the surprising properties discovered is that, when doped with an alkali metal (M), C_{60} becomes conductive at the M_3C_{60} state, and as it is further doped to the M_6C_{60} state, it returns to being nonconductive.[2,3] This behavior is easily understood in terms of the known molecular orbitals of the C_{60} molecule. Figure 2(a) shows part of the molecular orbital energy diagram for C_{60}.[4]

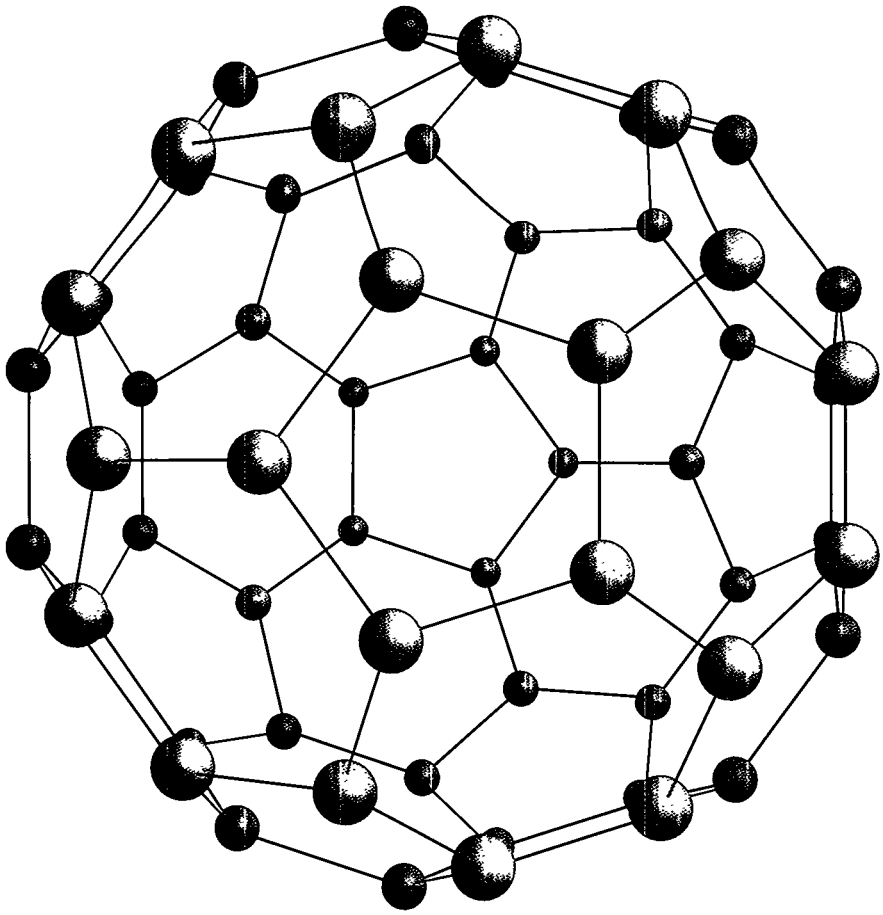
As C_{60} is doped by an alkali metal, such as lithium, electrons are transferred to the C_{60} molecules, where they populate the T_{1u} orbital. As the molecular orbital becomes half-filled by the transfer of three electrons from three alkali metal dopant atoms, shown in Fig. 2(b), the conductivity of the solid state increases owing to the mobility of these electrons in the conduction band formed by these molecular orbitals.

As the doping increases past half-filling, the conductivity of the material drops because of the

continued filling of the conduction band. Finally, when six electrons have been transferred, the "fully doped" state in Fig. 2(c), the band is filled and the material becomes nonconductive with a calculated band gap of 0.5 to 1.0 eV.[3]

Use of doped C_{60} in neutron detection

The existence of a highly doped semiconducting phase is of interest because it may be used in the design of a novel solid-state neutron detector. Such a detector would be based on the material $(Li^6)_6C_{60}$, expected to be semiconducting. The density of lithium in the materials would be about one atom per 120 Å,[3] resulting in about the same cross-section for thermal-neutron absorption as current ^3He-based neutron detectors. Because the material is solid state and would operate by measuring charged carriers produced by incident neutrons, deficiencies of existing detectors such as vibration sensitivity and poor discrimination with respect to gamma radiation should be reduced and/or eliminated. We are conducting measurements to determine the electronic characteristics of lithium-doped C_{60}. Progress is being made toward the initial neutron detection test using this material.



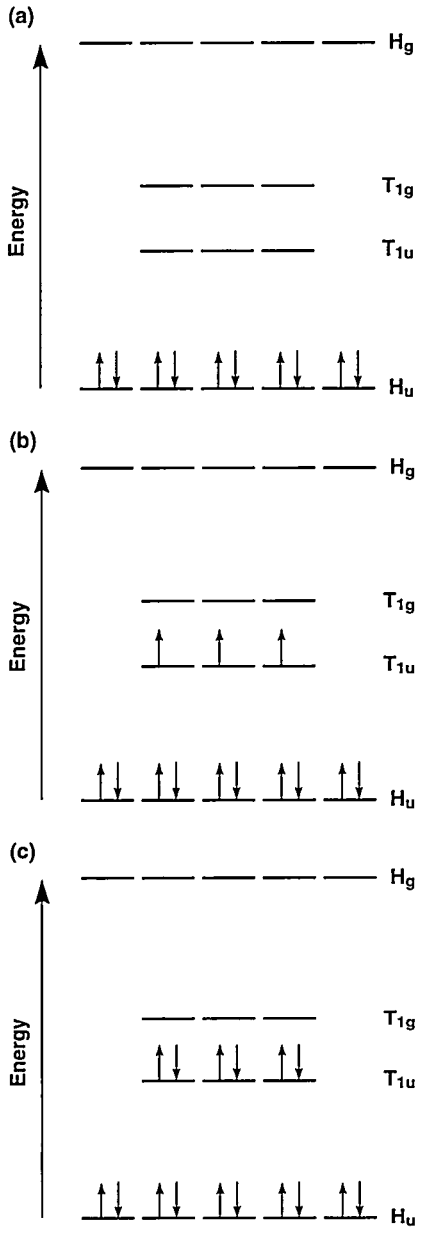
□ *Figure 1. The molecular structure of C_{60}.*

Our new synthetic route

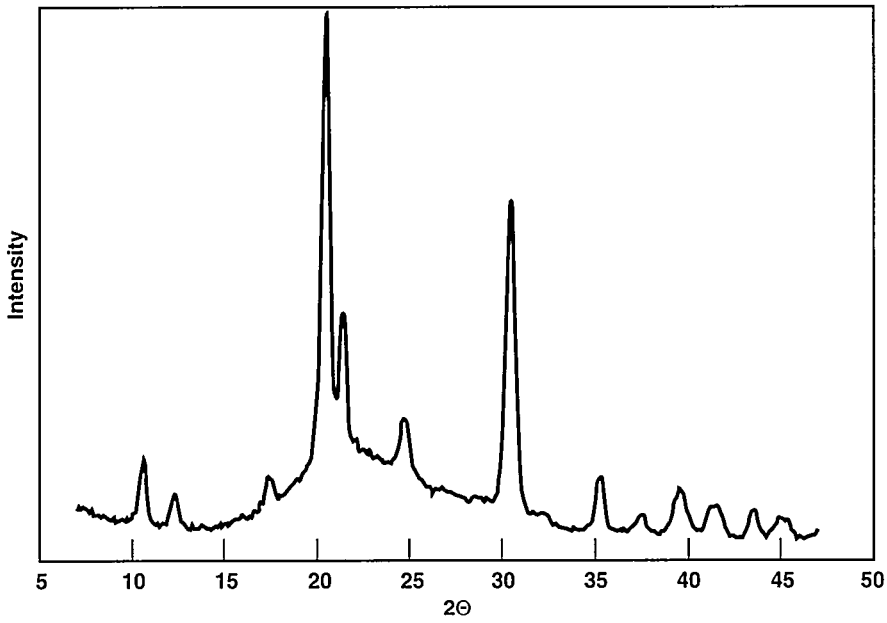
A technical challenge that must be met before a useful device can be made is the production in quantity of material of sufficient homogeneity and correct stoichiometry. The primary current synthetic production route is to expose films of C_{60} to alkali metal vapor. Control over the final stoichiometry is extremely difficult with this technique, and only a small amount of inhomogeneous material is produced.

We have developed a new synthetic route for the production of alkali-doped phases of C_{60}.[5] Alkali metals have long been known to dissolve in liquid ammonia to produce solvated alkali metal cations and solvated electrons. By weighing out stoichiometric amounts of alkali metal and C_{60}, and then introducing liquid ammonia, we produce a solution containing stoichiometric amounts of alkali metal cations and C_{60} anions. The desired doped phase is then obtained by removing the ammonia.

This synthetic route has been verified for Rb_3C_{60} by production of known alkali-doped superconducting phases. Figure 3 shows the x-ray diffraction pattern produced by Rb_3C_{60} made using this synthetic route. The pattern is characteristic of the known superconducting phase and shows no other impurity phases. AC mag-



☐ *Figure 2. The electronic configuration of (a) C_{60}, (b) C_{60}^{-3}, and (c) C_{60}^{-6}.*



☐ *Figure 3. X-ray diffraction pattern made by synthetically produced Rb_3C_{60}.*

netic susceptibility shows a strong diamagnetic signal indicative of a superconducting material with a transition temperature of 29.6 K. DC magnetic susceptibility shows both shielding and expected Meisner susceptibility responses. These data and others for both Rb_3C_{60} and K_3C_{60} verify the synthetic route. Typical syntheses have produced 0.3 g of material. This synthetic method should be scalable to produce any amount desired.

As of late 1994, we have applied our synthetic route to the production of Li_6C_{60}. We are in the process of studying the electrical properties of this material and using it in neutron-detector studies. ☐

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Miniature GC-MS for on-site chemical analysis

Brian Ruiz, John Bushman, Armando Alcaraz, David Tinoco, James Wong, John Cornish, Gerald Coutts, and Brian Andresen
Lawrence Livermore National Laboratory

Gas chromatography is an analytical tool that uses heated capillary tubing and an injection port to separate unknown organic chemical compounds into a vapor state that can be resolved and identified by a mass spectrometer.

A mass spectrometer is an analytical tool that contains an ion lens source, a mass analyzer, and a detector to identify the organic compound received from the gas-chromatography unit. The ion lens source converts the vapor into an electrical light beam. The mass analyzer filters the light beam into its individual mass components. The detector records the total ions for plotting on a graph. These organic compound mixtures are determined by breakdown of the compounds into their individual components and comparing them with a reference library of 100,000 chemical species.

Gas Chromatography–Mass spectrometry, or GC-MS, combines gas chromatography with mass spectrometry to rapidly and completely characterize the individual compounds in complex mixtures. GC-MS can precisely and exactly identify unknown organic chemicals collected, for instance, in environmental samples. This capability normally associated with bench-type laboratory instruments was miniaturized by LLNL's Forensic Science Center, as part of its nuclear nonproliferation research. LLNL's original miniature GC-MS instrument weighed 70 pounds, while other manufacturers' units were 150 pounds or heavier. LLNL transferred this technology to Viking Instruments Corporation. Viking builds instruments that weigh 150 pounds called SpectraTraks. The new joint venture currently under development created a 50-pound instrument.

CWC requirements for the GC-MS

LLNL was originally interested in designing portable analytical instruments because of the Chemical Weapons Convention (CWC). The CWC treaties require inspection teams to examine chemical processing facilities on-site to ensure treaty compliance.[1,2,3] During on-site inspections, samples from soil, water, and air can be collected and analyzed in real time to determine whether chemical-weapon agents, precursors, or byproducts are present. Because the inspection teams may encounter a wide variety of chemicals in the field while under tight time constraints, the GC-MS instrument must be as sensitive, robust, and accurate as commercial laboratory instruments.

A field instrument's requirements

To understand the constraints on miniaturizing a GC-MS instrument, it is important to recognize its requirements for the field. First, a field GC-MS requires all the same abilities as laboratory-sized equipment. It must analyze certain sets of chemicals in real time with prescribed accuracy. The chemicals of interest during a CWC inspection range from very volatile to semivolatile to polar compounds. A heated GC injector, a temperature-programmed gas-chromatographic column, and a heated ionization source can accomplish the timely chromatographic separation of volatile and semivolatile compounds.

Second, because most samples generated during the CWC on-site inspections are solvent extracts, the field GC-MS instrument must have an injector that can vaporize liquid samples. To

maintain the vapor phase for standard GC analysis, an injector capable of reaching 300°C is necessary.

Third, a gas chromatography column that can be programmed for temperatures ranging from ambient to very hot is highly desirable for real-world environmental samples. The column temperature slew rate that is programmable with a rate optimized for target chemicals also shortens collection delays when higher boiling-point unknowns are encountered.

Lastly, a field GC-MS instrument must be sufficiently sensitive to and have the mass range to detect and display all diagnostic molecular and fragment ions. The instrument must be accurately calibrated in the field. The ionization source must produce a stable fragment ion beam; therefore, the ion optics and mass analyzer should produce a reliable and reproductible mass spectrum that permits computer-assisted comparisons and identifications from a reference library.

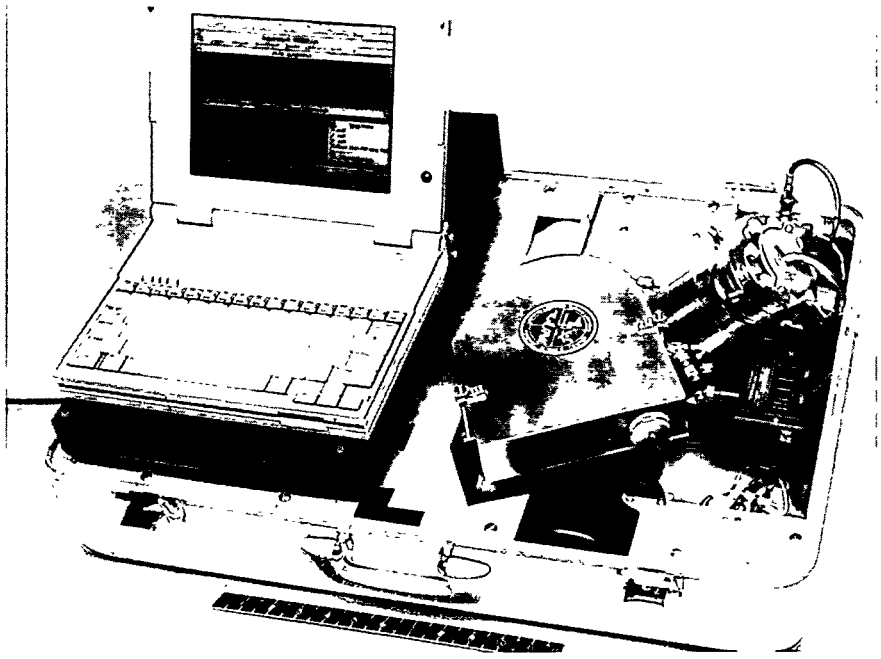
Portability

Finally, any portable, high-performance GC-MS system is governed by three parameters: power consumption, weight, and size. How much of each depends on second-guessing various scenarios of on-site inspections. The GC-MS instrument probably will be set up in a temporary staging area within sight of a chemical facility being inspected, possibly in an open field, within a car or truck, or on a van bed. Therefore, it is logical that the power setup should last at least eight hours before recharging or maintenance.

The CWC operational scenarios limit the weight. The package should be as lightweight and portable as possible with all the analytical capabilities of existing laboratory or transportable instruments (see Fig. 1). LLNL's standard suitcase-sized modules allow the inspection team to check the instrument as luggage, as it is most likely the team will travel by air. Hand-carrying the instrument also helps if travel time to the inspection site is short.

Performance

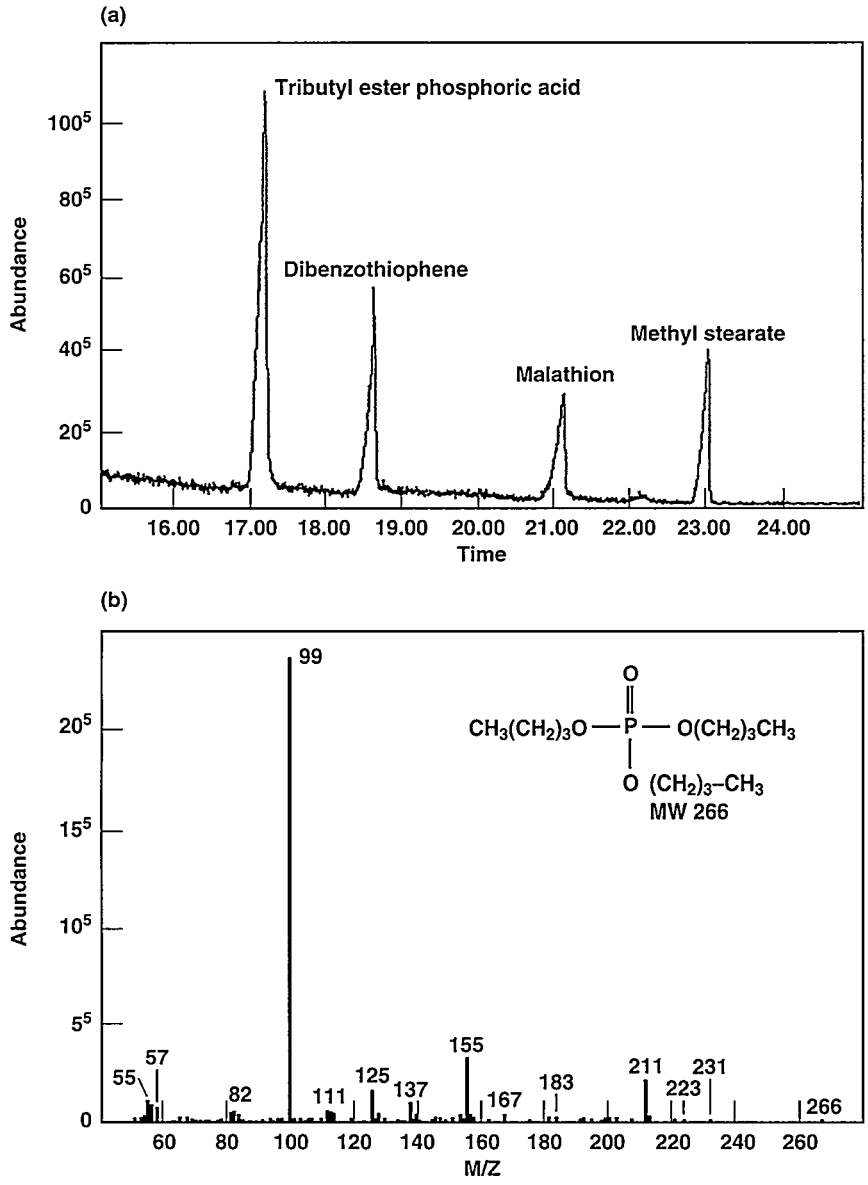
The analysis of three samples demonstrates the suitcase instrument's performance. The first test, Fig. 2(a and b), shows the total ion chromatography (TIC) and a mass spectrum of one component from an injection of 1 mL of an LLNL test standard. Figure 3 shows similar results appearing in a second test, an identical injection into a



☐ *Figure 1. A small, portable GC-MS instrument contained in a standard 9.5 x 18 x 27-in. suitcase. The package contains a GC, MS, vacuum system, 486 computer, and support electronics. As shown, it weighs 69 lbs. We are modularizing the instrument and its associated equipment and packing so that users can select the modules appropriate to their analytical requirements, size, weight, portability, and use in the field.*

5988 Hewlett-Packard GC-MS instrument. The relative peak areas and heights agree within a factor of 2. In a third test, for sensitivity, a 100-ng injection of methyl stearate was injected into the instrument; we estimate the sensitivity to be better than 1 ng/mL.

Early laboratory experience and field testing showed two problems: the cast aluminum vacuum housing of an early prototype was difficult to work with, and the vacuum integrity was unreliable. Early operational limits[4] led us to improve several parameters. Improved turbomolecular pumps will enable teams to use the instrument for nine hours in the field before recharging (which can be accomplished overnight). Some 200 injections depending on the sample preparation (typically 50 injections with the least sample preparations) can be made during the nine hours in the field. We installed a different vacuum enclosure, a new "getter" pump that improved the operational lifetime to longer than 31 days.

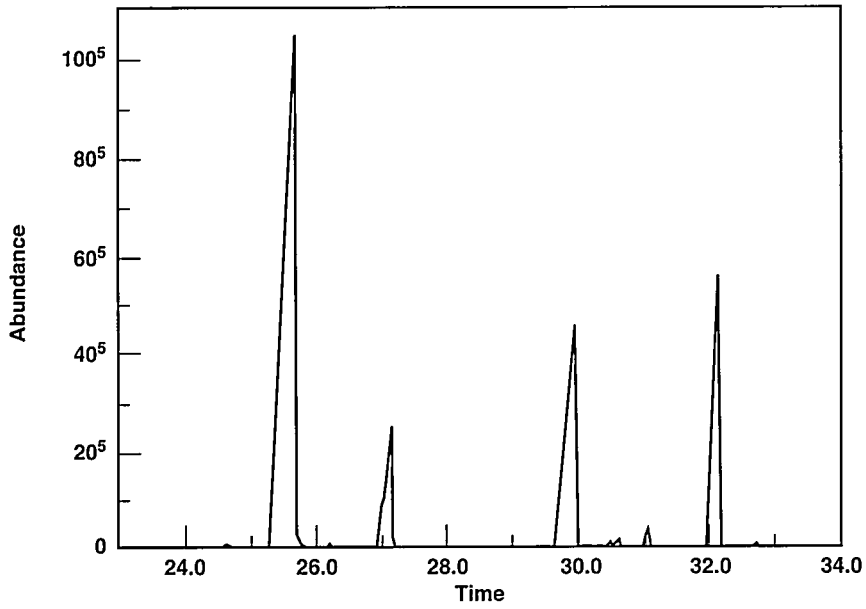


□ Figure 2. Total ion chromatogram and selected mass spectrum of a pesticide standard mix of four chemical components. The selected mass spectrum is of the first compound, tributyl ester phosphoric acid. The carrier gas for this sample was hydrogen with a back pressure of 4 lb. A full 1-μL solvent sample and standard compounds were injected into the suitcase GC-MS instrument.

Practical considerations

The number and type of samples and solvents injected into the instrument will determine some length of time (typically 30 minutes for ranges from 0–650 atomic mass units) the GC-MS instrument can operate in the field. Calculations suggest that the pumping charge at 235 g should allow 100,000–1,000,00 sample injections, depending on the solvent and sample split ratios. During a lifetime study, a total solvent load equivalent to 500 injections was sucessfully pumped by the new getter system. Our calculations indicate that the new vacuum system configuration can handle injections of 100 times the normal solvent load expected in an on-site inspection.

Besides its application in treaty compliance, the GC-MS instrument's operational lifetime, weight, and size also make it an ideal tool for firefighters and other emergency-response personnel to identify chemical spills, potentially hazardous emissions from warehouse fires, arson investigations, and other forensic identifications. □



□ *Figure 3. Total ion chromatogram of the same pesticide standard used to generate the results in Figure 2. To obtain these data, a full 1-µL injection was made into a Hewlett-Packard 5988 GC-MS table-top instrument. Note the similarities and peak shapes seen with the suitcase GC-MS results shown in Figure 2.*

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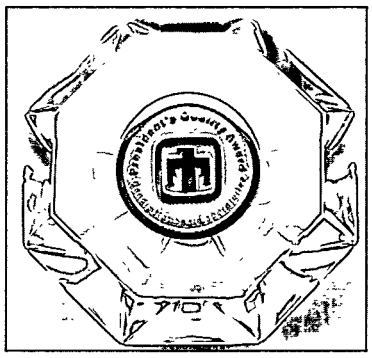
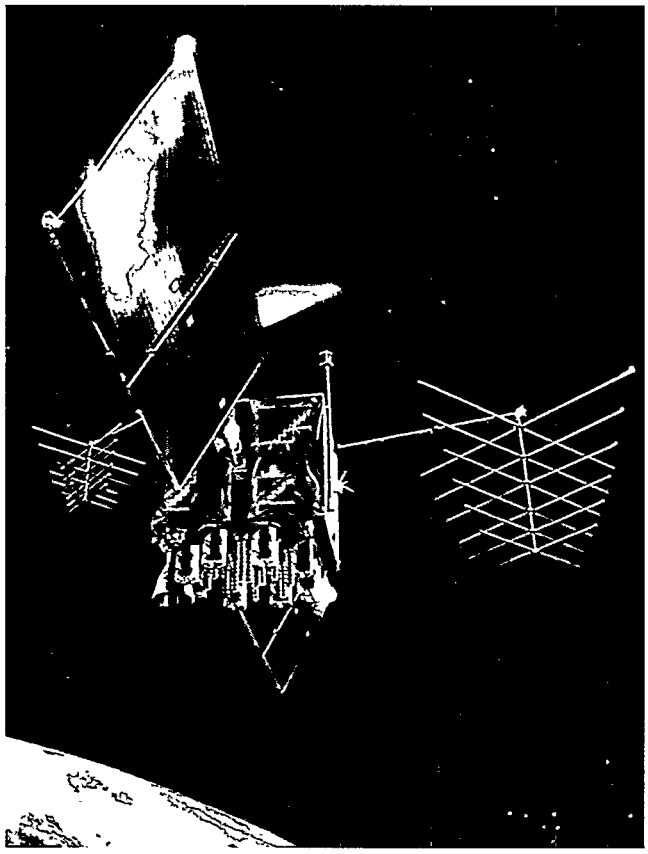
Winner of Sandia President's Quality Award

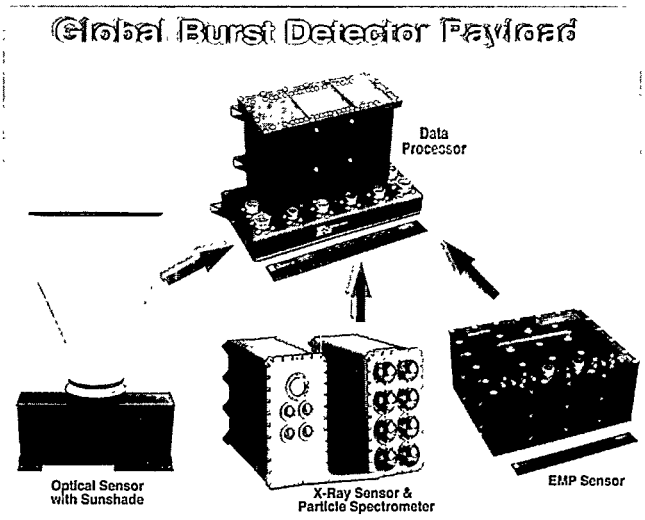


One Silver award winner in the 1994 Sandia President's Quality Awards was the global positioning system (GPS) satellite global burst detector (GBD). Team members were Norm Blocker, Paul Phipps, Steve Yearout, and Greg Christiansen.

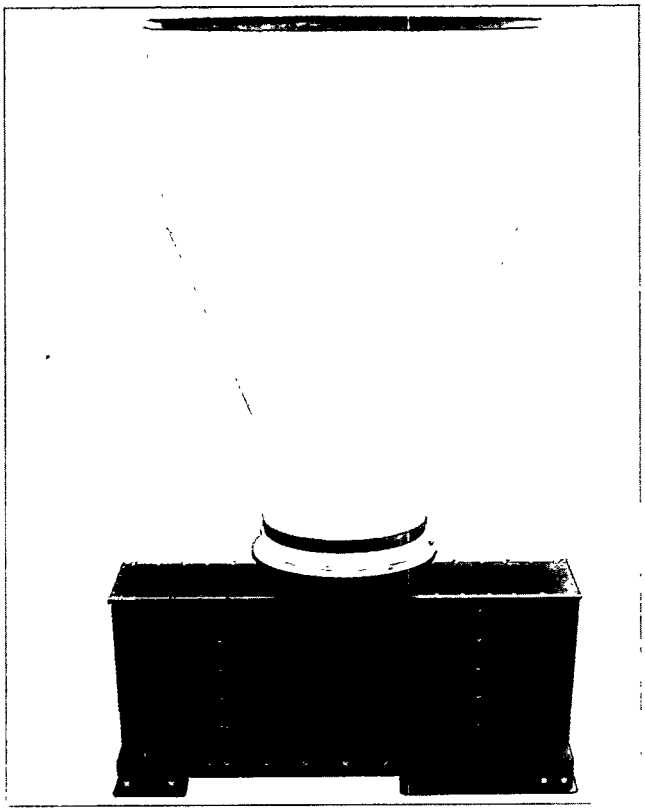
The nuclear detonation detection system (NDS) payload on the GPS satellite provides survivable worldwide surveillance and verifies compliance with the nuclear test ban treaties for the atmosphere and space. □



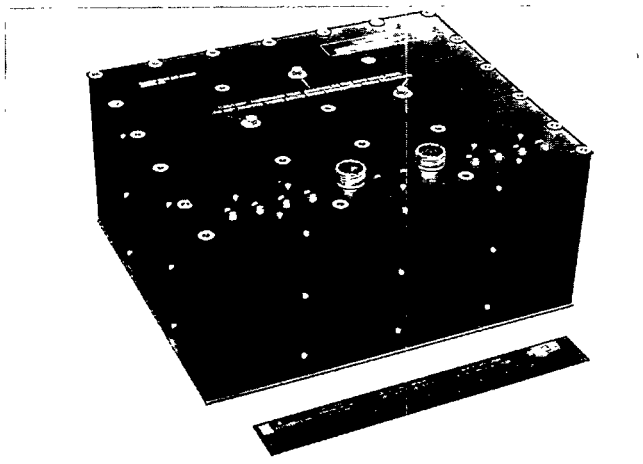
□ *Photo 1: An artist's concept of the GPS satellite.*



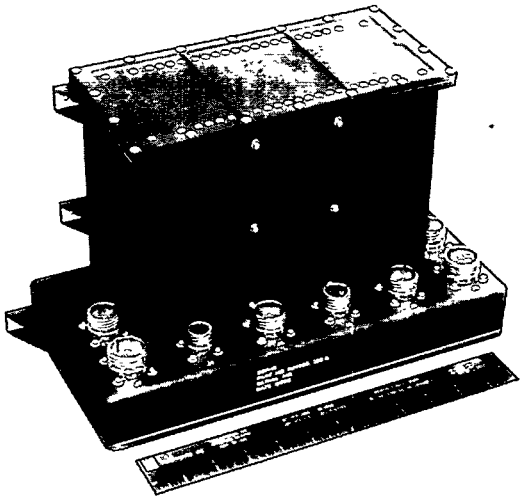
□ *Photo 2: The global burst detector (GBD) payload, which consists of a variety of sensors and supporting logic subsystems and testers.*



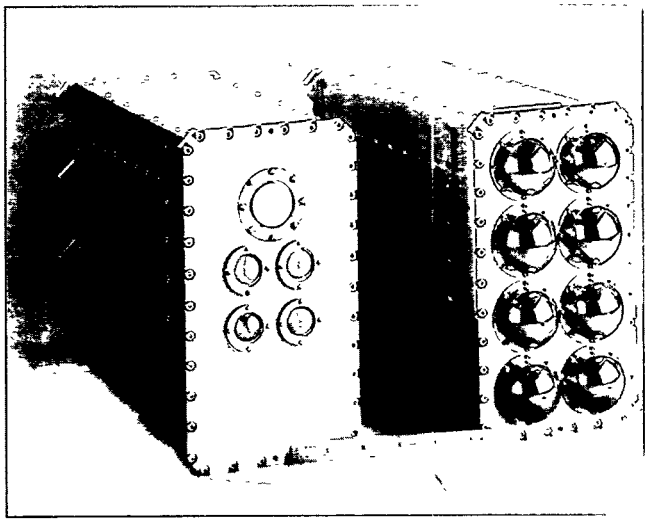
☐ *Photo 3. The optical sensor is a non-imaging radiometer (bhangmeter) that continuously observes the full-earth disk and responds to the optical signals generated by an atmospheric nuclear detonation. A large, prominent sunshade protects the sensor from sun blinding.*



☐ *Photo 4. The electromagnetic pulse (EMP) W-sensor detects EMP generated by the atmospheric nuclear detonations.*



☐ *Photo 5. The burst detector processor (BDP) processes most NDS functions, including nuclear detonation data. The BDP provides interfaces between the NDS payload and other satellite subsystems; conditions and routes power to the NDS sensors; processes commands and timing information; processes, formats, and stores data; and time tags nuclear events.*



☐ *Photo 6. The x-ray sensor at left is a four-channel x-ray detector that detects nuclear detonations outside the atmosphere by sampling the x-ray energy spectrum in four spectral bands. A particle spectrometer (shown at right) is used on approximately every sixth satellite to measure ambient electron and energetic ion (proton) fluxes in the operating environment of the satellite.*

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