GA-C22131 (20/97)

E-SMART SYSTEM FOR IN-SITU DETECTION OF ENVIRONMENTAL CONTAMINANTS

QUARTERLY TECHNICAL PROGRESS REPORT

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Work Performed Under
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U.S. Department of Energy
Idaho Operations Office
Idaho Falls, Idaho

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GENERAL ATOMICS PROJECT 3719/4450
August 1997

GENERAL ATOMICS

Contents

Abstract

General Atomics (GA) leads a team of industrial, academic, and government organizations in the development of the Environmental Systems Management, Analysis and Reporting neTwork (E-SMART) for the Defense Advanced Research Project Agency (DARPA), by way of this Technology Reinvestment Project (TRP). E-SMART defines a standard by which networks of smart sensing, sampling, and control devices can interoperate. E-SMART® is intended to be an open standard, available to any equipment manufacturer. The user will be provided a standard platform on which a site-specific monitoring plan can be implemented using sensors and actuators from various manufacturers and upgraded as new monitoring devices become commercially available.

GA's TRP team members include Isco, Inc., Photonic Sensor Systems, Inc. (PSS), Georgia Tech Research Institute (GTRI), Secor, Inc. (SECOR), Biode, Inc (Biode), Rapid Clip Neural Systems, Inc. (RCNS), Applied Research Associates, Inc. (ARA) and the U.S. Air Force Armstrong Laboratory Environics Directorate at Tyndall AFB(AL). Government interests are also represented by program managers from DARPA, DOE EM-53, and DOE-Idaho.

This DARPA TRP project will further develop and advance the E-SMART standardized network protocol to include new sensors, sampling systems, and graphical user interfaces. Specifically, the E-SMART team will develop the following three system elements:

A new class of smart, highly sensitive, chemically-specific, in-situ, multichannel microsensors utilizing integrated optical interferometry technology,

A set of additional E-SMART-compatible sensors and samplers adapted from commercial off-the-shelf technologies as well as a developmental sensing technologies contributed by Biode, Inc. and Sawtek, Inc.(see below), and

A Data Management and Analysis System (DMAS), including network management components and a user-friendly graphical user interface (GUI) for data evaluation and visualization.

General Atomics has signed Articles of Collaboration with another DARPA TRP awardee, Sawtek, to develop an E-SMART-compatible version of the Sawtek Intelligent Modular Array System (IMAS) for monitoring volatile organic chemicals (VOC's) in the environment. This collaboration will simplify the network development required to field the IMAS sensor, and will encourage the adoption of the E-SMART standard by increasing the number of commercially available E-SMART sensors. The Sawtek team now also includes Perkin-Elmer.

Figure 1 summarizes the vision and goals of the E-SMART TRP project.

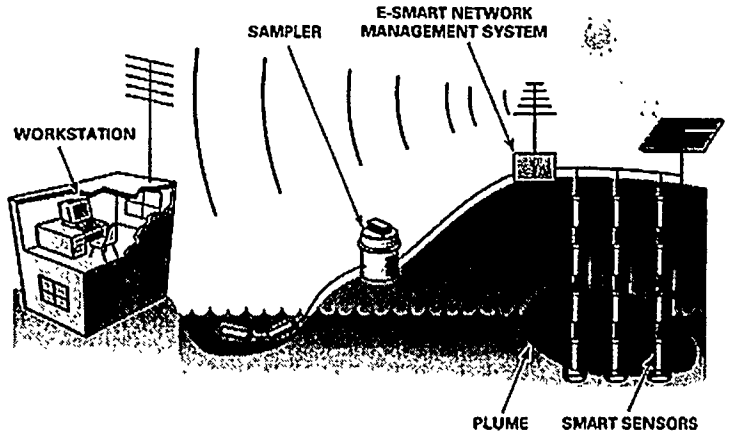
E-SMART TRP

TEAM

- **General Atomics** (team leader)
- **Isco, Inc.** • **Photonic Sensor Systems, Inc.**
- **Secor, Inc.** • **Georgia Tech Research Institute**
- **Armstrong Lab. Environics Directorate (USAF)**
- **DARPA** • **DOE EM-53** • **DOE-Idaho**
- Tinker AFB (OC/ALC-EMR) (USAF)*
- Rapid Clip Neural Systems, Inc.
- Biode, Inc. • Applied Research Associates, Inc.
- IMAS TRP Team (Sawtek, Inc - team leader)

(bold = original team member; * E-SMART TRP field test site)

VISION



OBJECTIVES

- **Develop and promote E-SMART®, an open standard for networking smart sensors at environmental sites.**
- **Develop and field test new, chemically-specific, multichannel smart sensors for detecting contaminants in air and water.**
- **Integrate commercially-available technologies into E-SMART devices designed to facilitate environmental sensing and environmental remediation process control.**
- **Develop a Data Management and Analysis System (DMAS), including network management components and a user interface for data evaluation and visualization.**

SCHEDULE

Task Name	1995				1996				1997				1998	
	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2
100 Development														
100.100 Microsensors														
100.200 E-SMART Integration														
100.300 Visualization														
200 Field Testing														
Preliminary Testing														
Integrated Testing														
300 Project Management														

Fig. 1. Quad Chart Summarizing the E-SMART Project

Executive Summary

Program Management

- An additional subcontractor, Applied Research Associates (ARA) was was added to the project to develop an E-SMART-compatible Multiplexer/ Data Logger.

Multichannel Microsensor

- Three invention disclosures were submitted to DOE by PSS/GTRI relating to the sensing of volatile organics with thin-film polymers.
- A method was devoped for patterning BTEX-selective polymer coatings on the E-SMART microsensor integrated optic detector chips.
- A basic pattern recognition scheme was identified for extracting individual BTEX concentrations from an array of BTEX-sensitive polymers. Rapid Clip Neural Systems is proceeding with reducing this scheme to a software algorithm.
- A trial integration of a first-issue E-SMART microsensor head is being machined and will be mated with a prototype E-SMART microsensor controller being fabricated by Isco next quarter.

Sawtek Integrated Modular Array Sensor (IMAS)

- Preliminary E-SMART electronics information was sent to Sawtek as requested.

Other Sensors and Actuators

- Applied Research Associates, Inc joined the team and is developing an E-SMART Digital Multiplexer/Data Logger
- An improved E-SMART sensor housing is under development to transition the original proof-of-concept design to a commercial version. First-issue housings are expected to be delivered in July.

E-SMART Network Management

- A software development strategy has been finalized for the next version of DMAS software. Specifications are now being developed.

E-SMART Field Testing

- Analysis of data being collected by the E-SMART network is continuing. The data rate was reduced. Maintenance of the network continues at a low tempo pending resolution of Tinker's continued participation and DOD/DOE funding vehicles for continued work.

Dual Use and Commercialization

- Armstrong Laboratory continues to explore expansion of E-SMART installations and demo sites.
- Armstrong Laboratory and General Atomics briefed the Air Force Center for Environmental Excellence at Brooks AFB on the E-SMART program in April.
- E-SMART was registered as a trademark by the United States Patent and Trademark Office

1. Work Conducted April - June, 1997

1.1 Multichannel Microsensor (PSS/GTRI)

WBS 100.110 - Develop Requirements and Design Configuration

A final design review for the E-SMART microsensor head was held in Atlanta on May 13th, with General Atomics, Isco, GTRI and PSS in attendance. Comments from the review have been incorporated in the design, and final drawings have been made. The microsensor head is 6.5 inches long and fits into a 1.5 inch diameter circular housing. To minimize complications from thermal effects, the microsensor head will be machined from invar. The microsensor head design is now complete.

WBS 100.120 — Develop Coatings & Identify Interferants

Three invention disclosures related to the sensing of volatile organics with thin-film polymers were submitted to the Department of Energy in accordance with contract provisions. The invention disclosures are titled: 1) "Polymer Laminates for Evanescent Field Sensors I: Polymer Overlays for Protection Selectivity and Increased Dynamic Range", 2) "Polymer Laminates for Evansecent Field Sensors II: Tailoring the Mechanism for the Desired Magnitude and Sign Change" and 3) "Reversibly Sensing Organics in Thin Films with Evanescent Field Sensors". A literature survey and patent search are being conducted to determine the patentability of these inventions.

WBS 100.130 — Design and Develop Glass Waveguides

A complete set of fabrication masks for the thirteen-channel E-SMART microsensor integrated optic detector chips were completed, and the first four detector chips were made.

The grating couplers and the Fresnel beam splitter/combiner elements in the initial chips were fabricated using a standard dry etch process. As previously reported, an attempt is being made to develop a less demanding deposition process for fabrication of these elements. Deposition fabrication of the grating couplers has been nearly perfected, but attempts to utilize depostion fabrication for the beam splitter/combiner elements have not yet been successful. It is likely that all of the detector chips needed for this program will be fabricated using dry etch processing

WBS 100.135 — Develop Sensor Protective Techniques

A method was developed for patterning BTEX-selective polymer coatings on the E-SMART microsensor integrated optic detector chips. Narrow sensing channels (1 cm long by 380 μm wide, with 190 μm gaps between channels) are defined using standard photolithography. The area outside the channels is functionalized with a fluorosilane, and a Teflon AF film is cast over the fluorosilane. Dissolved polymers are pipetted into the "chemical wells" formed by the mismatch in surface energy inside and outside the channels, and the solvent is allowed to evaporate. The hardened polymer coatings are then annealed in a solvent vapor to relax the coatings to a flat profile. (Coatings with curved profiles might act as optical lenses.) It was found that covering the channel surface with an

extremely thin, silane-derivitized polymer layer prior to addition of the sensing polymer layer significantly enhances the coating adhesion.

A thirteen-channel sensing polymer array was successfully patterned on the surface of a detector chip mock-up using this technique.

WBS 100.150 — Design Microsensor Electronics & Packaging

A basic pattern recognition scheme was identified for extracting individual BTEX concentrations from an array of BTEX-sensitive polymers. Concentrations will be deduced from a set of twenty-one independent variables comprising six phase shifts and the fifteen ratios between the six phase shifts. Rapid Clip Neural Systems is proceeding with reducing this scheme to a software algorithm. GTRI is continuing to generate calibration data for RCNS.

WBS 100.170 — Build & Test Prelim. Microsensor Model

A first-issue E-SMART microsensor head is being machined and fitted with a thirteen-channel integrated optic detector chip. A trial integration of this assembly with a first-issue E-SMART microsensor controller being fabricated by Isco will be attempted at a meeting in Atlanta on July 9th.

WBS 100.191 — Microsensor Support (Isco)

Continued discussions with GTRI regarding the present optical bench design and made additional modifications to accommodate a smaller housing. Did some initial investigation into modifications of the optical bench/housing design that would permit use of a round housing. Optical bench parts were entered and modified in Pro-E (CAD) as probe housing shape and size parameters were investigated. Did some work on the probe housing. The schematic for the Hitachi H8/3003 board was entered into the CAD system and the Hitachi electronics board layout was completed. All of the components for the board were ordered and received. CAD entry of the schematic for the Neuron circuit board was begun.

WBS 100.192 — E-SMART/LonWorks Interface (Isco)

Attended LonWorks Technology training at Motorola University in Schaumburg, IL on April 15-18, 1997. Clarified changes to the E-SMART standard.

WBS 100.193 — Embedded Software (Isco)

Experimented with low level code to read the TSL401integrated-circuit array sensor with the Hitachi H8/300 processor. The embedded software development was started using the IAR H8/300H c-compiler and PVCS version-control software.

WBS 100.194 — Probe with Electronics and Optics (Isco)

Worked on the schematics for the Lab prototype circuit boards. The schematic for the Hitachi H8/3003 board was entered into the CAD system and the board layout completed. All the components for the Hitachi board were ordered and received. CAD entry of the schematic for the Neuron circuit board was begun.

WBS 100.195 — PC-based E-SMART Software (Isco)

No activity this period.

WBS 100.196 — Lab/Local Testing (Isco)

No activitiy this period.

1.2 Sawtek Integrated Modular Array Sensor (IMAS)

Sawtek previously asked for input from GA on E-SMART SDP form factors and description. Preliminary inputs were prepared and were sent to them.

1.3 Other Sensors and Actuators (GA, Biode)

WBS 100.200 — E-SMART Integration

The pH glass electrode sensor was added to the E-SMART field demo site in March. This electrode was connected to the network via a dedicated 100 foot cable which ran from Monitoring Well 1-098 to a standard pH sensor signal conditioner inside the E-SMART trailer. A long cable was chosen to reduce the chance that a bad connector or interim connection would interfere with the low-level signal for the sensors. The signal conditioner current output was connected to a modified PSI/SDP board to form an E-SMART node within the trailer. A ground loop problem was overcome and data was collected from the sensor for approximately two months. Analysis of the data indicated a variation of roughly ±0.1 pH units on a diurnal cycle. This variation may have been due to temperature changes within the E-SMART trailer. A set of additional sensors were installed in the trailer to monitor temperature variations. The pH sensor also appeared to have drifted by approximately 0.1 pH units between May 6th and June 5th which may be due to electrode poisoning. The glass electrode did not appear to be fouled after the two month immersion.

Work continued on improving the E-SMART sensor housing. The new sensor housing design will transition the original prototype design to a manufacturable version. A major improvement is the elimination of the potting process for sensor components, which will make E-SMART sensor fabrication quicker and easier. Another improvement realized by the new design will allow transducers to be more easily interchanged.

Biode Work Effort:

BIODE's related SBIR research has focused on the development of frequency-based piezoelectric mass sensors for mercury ions and various biochemicals in aqueous solutions.

Piezoelectric Transducers:

Quartz transducer effort is yielding stable baseline data and static solution mercury detection at or near the Safe Drinking Water Act (SDWA) limits. Hybrid electrochemical / piezoelectric sensors detected 10 ng Hg in 600 microliters of liquid. Signal to noise indicates ~2.4 ng/ml sensitivity (2 is SDWA.) Additional measurements of quartz SHAPM transducers provide measurable responses to electrochemical excitation in the presence of mercury and none without mercury.

Lithium tantalate transducers have exhibited sensitivity comparable to quartz devices. Temperature stability is worse; however, size and electrical efficiency are improved.

Other transducer structures are being compared using both materials. Quartz prototypes of these alternate structures have been measured and require further refinement for proper electrical interfacing. Lithium tantalate prototypes of these alternate structures have been built. Experiment and theory did not agree in either of two design cycles. The lithium tantalate material is suspected to have unique material properties, which could offer substantial design benefits if properly accounted for.

Instrumentation-related effort included:

BIODE is designing printed circuit board prototypes of several oscillators, which convert the sensor's mass loading data into measurable frequency data. The oscillator performance is primarily determined by the piezoelectric sensor element but is also dependent on the components and structure of the circuit. A frequency-counter ASIC has been designed and prototypes are being manufactured. This chip interfaces four sensors to the microprocessor. BIODE is also designing electrochemical control circuit boards as pre-prototypes of a potential second ASIC. These boards are currently being tested.

Microcontroller Programming:

BIODE designed a 16-bit microcontroller system using Motorola's 68HC916X1 microprocessor. This controller is intended for low-end data collection and reporting requirements where little local data processing is required. The effort to-date has included conceptual design and the assembly and testing of major subsystems in both hardware and software. User interface basics have been coded for electrochemical waveform control. Serial port communications has been coded between a PC and the sensor. This step is the first in a progression towards the use of Neuron chips for LonTalk / E-SMART integration of the sensor controller.

1.4 E-SMART Network Management (GA)

WBS 100.230 — E-SMART Network Management

Development of the next version of the E-SMART DMAS software continued. A program development approach was chosen and specifications for the program are being written.

1.5 Field Testing (GA, SECOR)

WBS 200 — E-SMART Field Testing

The Tinker AFB field test is sponsored in part by the Air Force Air Logistic Command in Oklahoma City, Oklahoma (OC-ALC) under contract number F34601-95-D-0374, Task Order 14, SDRL A025. The goal of the testing is to deploy an E-SMART network in the Southwest Tank Area (SWTA) site, to evaluate system performance, and to identify opportunities for system improvements. The E-SMART network has a graphical user interface tailored to the SWTA site and allows OC-ALC Environmental Management & Restoration (EMR) Division personnel remote access to data from the site.

During the quarter a few sensors were inspected and removed from the system for visual inspection and post test examination after faults were detected in their performance. With the exception of the addition of a pH sensor and additional temperature sensors, no major changes were made in the network installation pending resolution of negotiations with Tinker regarding either extension of the field test or disposition of the equipment. Data continues to be logged. Additionally, startup of the "Bioslurper" system at the SWTA is expected next quarter and will complicate data analysis due to its pumping action.

1.6 Visualization

WBS 100.300 — E-SMART Visualization (GA)

Visualization activities will be subsumed within the development of the DMAS system for the duration of the project. This activity will be reported under E-SMART integration activities.

1.7 Program Management (GA)

WBS 300.100 — E-SMART Program Management

Applied Research Associates, Inc. was given a $25k subcontract and will contribute $25k of cost share to the team to produce an E-SMART-compatible multiplexer/data logger unit.

1.8 Dual Use and Commercialization (GA, Armstrong Lab)

WBS 300.200 — Dual Use and Commercialization

Armstrong Lab has identified several potential E-SMART demonstration sites in addition to Tinker AFB. Candidate sites are Port Hueneme, CA (USN), the Puget Sound Naval Shipyard (USN), and the Groundwater Restoration Field Laboratory (GRFL), Dover AFB, NJ. They also arranged an E-SMART briefing at Brooks AFB in April for the Air Force Center for Environmental Excellence (AFCEE) and the Armstrong Laboratory Occupational Health and Safety Group.

E-SMART® was issued as a Registered Trademark in May by the United States Patent and Trademark Office.

Preliminary work began on setting up an E-SMART Standards Organization to promote E-SMART devices and maintain the E-SMART standard.

2. Problems

Isco continued to have internal problems generating required monthly expense reports and SF270 invoices. These problems were aggravated by the end of the fiscal year workload at Isco involving numerous reporting requirements to outside auditors and regulatory agencies. GA worked with Isco to solve the problem and expects that Isco will be current by the middle of August when a member of the GA finance staff will visit them.

3. Plans for the Next Quarter

- Team-member development activities will continue.

- GA hopes to issue the next revision of the E-SMART Node Specification.

4. Milestones and Deliverables

E-SMART System for In-situ Detection of Environmental Contaminants - Quarterly Technical Progress Report, Quarter 1, Calendar Year 1997 - completed and delivered per contract requirements.

Financial Status Report-Standard Form 269A, Reporting Period 4/1/97-6/30/97 - completed and delivered per contract requirements.

5. Papers and Conferences

- Isco personnel attended Lonworks training at Motorola University in April 1997.

6. Financial Status Report

Per contractual direction, DOE form SF-269A, "Financial Status Report", has been completed by GA for this quarter and has been distributed to the following individuals at DOE-Idaho:

- Patrick Trudel, Program Manager
- Chief Financial Officer, Financial Management Division
- Rebecca Rich, Accounting, Financial Management Division
- Wade Hillebrant, Contract Specialist

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