REMOTE SENSING LABORATORY

*Operated by Bechtel Nevada for
the U.S. Department of Energy*

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FINAL PROJECT SUMMARY REPORT

BECHTEL NEVADA AND FCI ENVIRONMENTAL, INC. COOPERATIVE RESEARCH AND TECHNOLOGY DEVELOPMENT PROJECT

OCTOBER 1, 1995, TO SEPTEMBER 30, 1998

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Las Vegas, Nevada

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Richard J. Pollina, Ph.D.
Science Specialist

ABSTRACT

This is a report summarizing work on a small project dedicated to adapting a new chemical sensing platform for the U.S. Department of Energy and its customers. At the same time and in the spirit of technology transfer, FCI Environmental, Inc., would receive technical support in the form of expertise from the U.S. Department of Energy to assist in developing this product. The sensor is a hybrid integrated-circuit, optical waveguide, chemical sensor that is patented by FCI Environmental, Inc., and manufactured under license by Texas Instruments, Inc.

A "down-hole" penetrometer probe was designed for use in hot, 60°C, hydrocarbon-saturated, saline environment at a depth of 200 feet. The probe design includes three chemical sensing, hybrid integrated-circuits with chemical reference and measurement channels, a water seal, output electronics, and a removable measurement head for replacement in the field. A hand-held chemical detector prototype—containing a hybrid integrated-circuit chemical sensor with reference channel, user alarm, and level display—was designed and constructed, and a software interface developed to operate the hand-held sensor interfaced with a laboratory data acquisition system.

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PROJECT SUMMARY

In the performance of this project, Bechtel Nevada used the hybrid chemical sensor patented by FCI Environmental, Inc., and designed a three-sensor probe that can be used downhole in hot saline water. Bechtel Nevada also designed and built the first concept prototype (Mark 1) of a hand-held chemical detector. Based on this prototype, both FCI Environmental, Inc., and the U.S. Department of Energy (DOE), Office of Nonproliferation and National Security, Office of Research and Development (NN-20), jointly funded a working prototype (Mark 2) of the hand-held chemical detector that has generated interest from commercial customers and the U.S. Customs Service as well as the Environmental Protection Agency (EPA).

The final step in the project, completed in fiscal year (FY) 1998, demonstrated the ability to connect the chemical sensing integrated circuit (IC) to a computer using National Instruments' "Labview" software, a data acquisition standard that would significantly enhance the usefulness of the sensor in data acquisition applications. Completion of this project was delayed until FY 1998 because the U.S. Department of Energy (DOE) national laboratories were setting requirements for a sensor-communications interface. These requirements were subsequently canceled. "Labview" became the only viable choice, and work resumed on the project.

GOALS

The goal of this project was to provide technical improvements by incorporating the chemical sensor in a useable device, perhaps even as part of an existing commercial system, while adhering to a cost model of less than \$500 per unit (in quantities of 10,000). These improvements were in the form of schematics, mechanical drawings, and prototypes. The key was to enhance system capabilities as efficiently and effectively as possible.

WORK STATEMENT

In this project, a new detector was designed and built based on a hybrid IC, optical wave-guide, chemical sensor developed by the commercial partner. DOE expertise was utilized to develop an improved sensor system that was cost-effective and more versatile than the existing systems and used standardized interfaces so that future add-ons were made compatible. Since this was a cooperative research and development effort, the deliverables represented the best effort of both FCI Environmental, Inc., and DOE in applying DOE technology for the mutual benefit of both parties.

The sensor probe system was designed for ready deployment in typical field applications, which required the system to be modular, ruggedized, and extremely stable. The sensor-head interface made the unit capable of using multiple sensors while the output interface allowed the sensors to communicate with various output destinations, such as data logger, personal computer, or radio frequency communications module.

Additionally, FCI Environmental, Inc., developed a down-hole sensor system that was currently available on the commercial market. FCI Environmental, Inc., continued to develop this system and adapt the down-hole sensor system to the new sensor, thereby providing a superior product

for their customers. This cooperative research and development effort utilized DOE expertise and provided possible solutions for several of the technical hurdles FCI Environmental, Inc., faced.

The following two issues represented the major areas of technical interest:

System Configuration

The requirement was to develop a method that would allow three or more sensors in a single sensing head to be connected to a single processing unit. The design allowed the system to be configured under "field" conditions.

Smart Sensor

To incorporate multiple sensors into a single processing unit, the sensors carried identifying information and signal-conditioning circuitry. These factors adjusted the sensor output to compensate for calibration constants, scaling factors, or other data particular to the individual sensor. The data and circuitry were located in the sensor head. This arrangement allowed different types of sensors to be connected in the field making it necessary to reconfigure the processing and measurement sections.

DELIVERABLES

The deliverables to FCI Environmental, Inc., included schematics, mechanical drawings, and hardware as deemed necessary by the cooperating organizations. The deliverable to DOE was a final report detailing the accomplishments of the project, which also included a letter from the commercial partner detailing the results achieved through this cooperative development and technology effort.

WORK ACCOMPLISHED

FY 1996 A "down-hole" penetrometer probe was designed in FY 1996 for use in hot, 60°C, hydrocarbon-saturated, saline environment at a depth of 200 feet. The probe design included three chemical sensing hybrid ICs with chemical reference and measurement channels, water seal, output electronics, and removable measurement head for replacement in the field.

FY 1997 The Mark 1 hand-held chemical detector prototype—containing a hybrid IC, chemical sensor simulator with reference channel, user alarm, and level display—was designed in FY1997 and constructed. Two papers pertaining to the Mark 1 were presented, one at the Fifth International EPA Symposium, Las Vegas, Nevada, January 29–31, 1997, and one at the European Symposium on Environmental Sensing III: Conference on Environmental Fibre Sensors and Application, Munich, Germany, June 16–20, 1997 (see Attachment C). Work on the Mark 1 prototype was funded by DOE Defense Programs . DOE/NN-20 funded fabrication of the Mark 2 prototype.

FY 1998 A National Instruments' "Labview" software interface for the hybrid IC was

developed. The purpose of the “Labview” interface was to enable testing of sensor coatings in the laboratory before they were installed in DOE devices. The interface also permitted the testing of DOE laboratory-developed sensor coatings to the benefit of both DOE and FCI Environmental, Inc. During late FY 1997 and FY 1998, DOE/NN-20 funded the Mark 2 functional prototype based on Mark 1 results. Mark 2 advanced the development of the hybrid IC chemical sensor a step further by adding a numerical display, temperature compensation, sensitive chemical sensor, carefully designed fan/sampling chamber assembly, and RS-232 output. A paper on the Mark 2 was presented at the Conference on Chemical Microsensors and Applications at the International Symposium on Industrial and Environmental Monitors and Biosensors, Boston, Massachusetts, November 2–5, 1998 (see Attachment D).

The “Labview” module, developed in FY 1998, made it possible to read data from the hybrid IC chemical sensor with any Windows-based personal computer. This capability was demonstrated using the Mark 2 hand-held detector.

MILESTONES (with completion dates)

# Develop a plan detailing the allocation of resources	February 15, 1996
# Develop a design strategy for assembling a multi-sensor system. Through this effort, a chemical sensor head for a penetrometer probe was developed with a field-replaceable sensing head capable of measuring three different chemical species at once.	September 30, 1996
# Develop a design strategy for housing the multi-sensor system. Through this effort, the multi-sensor housing was developed to be durable in the down-hole environment yet permit the user to interchange the sensors without compromising the probe's immersibility. This effort resulted in a design that could be manufactured at a low cost.	September 30, 1996
# Develop a design strategy for a smart sensor system. A prototype design was developed for a hand-held chemical detector with user-adjustable, alarmed interface incorporating a visual readout as well as an audible and vibrating alarm.	September 30, 1997

#

September 30, 1998

Develop improvements in the electronics package.

A user inter-face based on commercially available products was developed to demonstrate the possibility of acquiring computer data both in the laboratory as well as in the field.

DISCUSSION—Benefits of Optical Waveguide, Sensor-Probe Systems

DOE benefitted from this project with the creation of a cost-effective, versatile sensor-probe system that can be used to detect and measure a large number of target species, including petroleum hydrocarbons, certain chemical agents, and heavy metals such as mercury, lead, and actinides. The sensor-probe system was designed to accommodate ready deployment in typical field applications; therefore, the system is modular, ruggedized, and extremely stable. No other hand-held sensor like this exists in the DOE system.

With this technology—perhaps more than with any other technology—DOE has a significant advantage in the capability of producing detectors at low cost (under \$500 in quantity). Unlike existing Surface Acoustic Wave (SAW)-based devices, which are heavily funded by DOE and the Department of Defense, this sensor can be used in both water and air or at their interface. The sensor uses commercially developed, hybrid IC technology provided by Texas Instruments, Inc., thereby significantly reducing procurement costs to DOE. Additionally, the modular sensor is capable of supporting a variety of sensing techniques including index of refraction, absorption, and fluorescence with both end-point and rate-of-change sensing.

Under this cooperative agreement, DOE developed a working familiarity with the hybrid sensor and is developing the sensor into one that can be used for specialized DOE needs. An example of an application is the DOE Pocket Pager,¹ produced for another DOE branch. In this case, the Pocket Pager becomes a personal dosimeter for hazardous materials by incorporating the hybrid chip into the pager. The IC is a new advance in applying technology that is of significant value to the DOE and its customers.

The second sensor platform produced from this project is the design and prototyping of a system capable of continuous operation in water to a depth of at least 100 feet—either salt water or "sweet" water. The sensor platform is capable of working in high, dissolved concentrations of the target specie. For example, in measuring petroleum hydrocarbons, the system is capable of operating continuously in water having saturated levels of gasoline (roughly 250–300 parts per million). Currently the only reliable sampling technique requires an individual to physically collect a sample at the testing site and then forward the sample to a laboratory for analysis. The probe can be operated remotely and continuously therefore eliminating the need for human interaction and sampling. This is a significant breakthrough in monitoring DOE sites. The design and prototype were completed successfully.

¹ Pollina, R.J.; Saini, D.; Klainer, S.M. "A Pocket-Sized Solid-State Optical Waveguide Chemical Sensor for In-Field Monitoring of Toxic Species" in the *Proceedings of the Fifth International EPA Symposium, January 29–31, 1997*. Las Vegas, Nevada; January 1997.

ATTACHMENT A

LETTER FROM FCIE TO BECHTEL NEVADA

FCI ENVIRONMENTAL, INC.

1181 Grier Drive, Building B, Las Vegas, Nevada 89119

(702) 361-7921 FAX (702) 361-9652
800-510-3627

March 16, 1999

Mr. John Mitchell, Jr.
General Manager
Bechtel Nevada
M/S NLV001
P.O. Box 98521
Las Vegas, NV 89193-8521

Dear Mr. Mitchell:

Over the past three years, FCI Environmental, Inc. (FCIE)—a subsidiary of FiberChem, Inc.—has greatly appreciated the opportunity to collaborate with U.S. Department of Energy and Bechtel Nevada in a cooperative research and development effort to expand advanced sensor technology. I am especially pleased to report that this very productive relationship, under the expert guidance of Dr. Richard Pollina, has resulted in two important advances in sensor technology based on FCIE's patented solid-state, hybrid integrated-circuit, optical waveguide, chemical/biochemical sensor (Sensor-on-a-Chip®).

A three-integrated-circuit sensor platform capable of achieving in-well/down-hole analyses to depths of 250 feet was successfully designed, and the initial design of a prototype hand-held sensor package was successfully constructed. Presently, versions of the hand-held sensor are being developed to detect hydrocarbons and polar solvents. Additionally, this work has led to an interest in evaluating sensor chemistries that are under development at the national laboratories for inclusion in the hand-held instrument.

Dr. Pollina has been instrumental in directing and coordinating the successful progression of this cooperative research and development effort. His technical knowledge and insight as well as his managerial focus complemented our project team and significantly contributed to a very favorable outcome.

FiberChem, Inc. was founded in 1989 for the sole purpose of developing, engineering, manufacturing and marketing optical chemical and biochemical sensors. It sells FOCS® (fiber optic chemical sensors) for the detection of hydrocarbons in water, air and at the water-air interface. The FiberChem technical staff are working on the development of advanced environmental and military sensors based on immunoassays, organometallics and designer polymers. FiberChem develops the complete sensor system, sense and reference chemistries and biochemistries, hardware and software, in order to assure the integrity of its products. Its next commercial undertaking will be a hand-held sensor package based on the subject cooperative research and development effort. FiberChem personnel have 22 U.S. Patents and 21 Foreign Patents in the area of optical sensors and several other patents pending.

FCI ENVIRONMENTAL, INC.

An FCI/FiberChem, Inc. Company

John Mitchell, Jr.
March 18, 1999
Page 2

FCIE values this joint association with the U.S. Department of Energy and Bechtel Nevada, and we look forward to continuing a successful collaboration to commercialize the pocket-sized chemical sensor system thus bringing this cooperative research and development effort to fruition.

Very truly yours,

Geoffrey F. Hewitt
CEO

cc: I.W. Ginsberg, M/S RSL-10
K.R. Lamison, Jr., M/S RSL-13
R.J. Pollina, M/S RSL-10
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ATTACHMENT B
SUMMARY PROJECT CHART

Cooperative Technology Development Program

Completed Design of 3-sensor Probe

Photo of sensor IC "chip"

POC: Dr. Richard Pollina, pollinrj@nv.doe.gov

Versatility of Probe Design

- Down-hole or penetrometer usage
- 60°C saline, depth to 250 feet
- Encapsulated, field-interchangeable head
- Only chemical sensing surfaces exposed
- Holds 3 or more chemical sensors
- Calibration data in sensor head
- Built-in microprocessor

Advantages of Sensor

- Integrated packaging
- Optical stability
- Active and passive operation
- Sense and reference channels included
- Suitable for special applications

Chemistry Developed to Date

- Alcohol (breath analysis)
- Cocaine, and atrazine immunoassay
- Petroleum hydrocarbons

DP-17

FiberChem, Inc.

Remote Sensing Laboratory

Bechtel Nevada

ATTACHMENT C

CONFERENCE PAPER

A FIELD-HARDENED, OPTICAL WAVEGUIDE, HYBRID INTEGRATED CIRCUIT MULTI-SENSOR CHEMICAL PROBE AND ITS CHEMISTRY

Richard J. Pollina, Ph.D.
Roger L. Himka
Devinder P. Saini
Stanley M. Klainer, Ph.D.

Presented at the European Symposium on Environmental Sensing III: Conference on Environmental Fibre Sensors and Applications, June 16–20, 1997, Munich, Germany.

A field-hardened, optical waveguide hybrid integrated-circuit, multi-sensor chemical probe and its chemistry

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ABSTRACT

A single probe containing three hybrid integrated-circuit, optical waveguide, chemical-biochemical sensors (chip sensors) has been developed. Each chip sensor contains two hybrid waveguides—one for sensing and one for reference. The sense waveguide is coated with a species-specific or group-specific chemistry or biochemistry. The reference waveguide is coated with a version of the sense chemistry or biochemistry, which is not sensitive to the analyte. The integrated structure is encapsulated and contains a single fixed light source, two detectors (reference and sense), and an optical train. The design is amenable to fluorescence, absorption, and refraction measurements.

The three chip sensors are individually mounted in a probe that contains all of the electronics and computing capability necessary to collect and process the output information from each chip sensor. Only the surface of the individual chips are exposed to the target analytes. The probe is rugged, intrinsically safe, and can operate under 75 m (250 ft) of water.

1.0 INTRODUCTION

Advances in chemical-biochemical sensors have led to a continuing interest in their use for environmental monitoring. These advances have been driven by the ever-increasing interest in protecting and remediating the environment and measuring the efficiency with which this is being accomplished. These exigencies include improved sensitivity, specificity, better precision, multiple analyte (pollutant) analysis, and small size. To meet these demands, a probe has been developed containing three hybrid integrated-circuit, miniature sensors capable of detecting and quantifying three different species in water and vapor.

The multi-sensor probe consists of the individual sensor chips with their species-specific or group-specific coatings and with the electronics and software to handle and process the response of the sensor chips. The probe is constructed to be waterproof to a depth of 75 m (250 ft) and to operate in the range of 0–50°C. The probe configuration is the key to a practical, commercially viable, multi-sensor package. The chip approach has been selected as most workable and versatile from both a design and implementation point of view.¹⁻³

2.0 INTEGRATED CHIP STRUCTURE

The optical waveguide chemical-biochemical sensor is the transducer in an information collection system that obtains *in situ*, real-time data about the presence and concentration of specific species or groups of compounds in chemical or biochemical systems.

Various types of sensing chemistries and biochemistries can be used including but not limited to inorganics, organics, polymers, metals, metal compounds, organometallics, bio-inorganics, enzymes, antibodies, and microbes. These can be immobilized mechanically or chemically using polymers, solgels, hydrogels, silanes, and organics. Measurement techniques are selected from absorption, refractive index, or fluorescence.⁴⁻⁶

The basic design for an optical chemical sensor is very simple: a light source, the optical waveguide with sensing chemistry attached, and the detector. In order to improve the performance characteristics as well as to simplify the manufacturing, the sensor has been reduced to a solid state, integrated, chip-level optical waveguide package (Figure 1). The chip package shown in Figure 1 incorporates a central LED for a light source, two waveguides (a sense arm and a reference arm), and matched photo detectors at either end of the package. The shaded areas are the two waveguides (sense and reference), the "V" in the center contains the reflectors which direct light into the waveguides, and the end pieces contain the individual detectors. The pins extending below the package provide all of the connections necessary to operate the sensor.

A cross section of the layout of the sensor platform showing the total integrated package is illustrated in Figure 2. Each sensor is an integrated structure with a semiconductor chip as the substrate and includes a fixed light source, multiple detectors (one for sense and one for reference), and an optical train. Optical waveguides are coated with sensing chemistries-biochemistries and attached securely to the "chip." The waveguide arms can be fixed on the substrate (e.g., by gluing) or can be removable. The waveguide arms, or parts thereof, can also be formed (e.g., molded) as part of the substrate or chip itself. The chemistries-biochemistries used for reference are usually an inert form of the sensing chemistry. These are attached to the second or reference waveguide. The basic mechanism of measurement using sensing and reference arms is a comparison of the output of the sensing and reference arms where the sensing arm is affected by the target analyte and the reference arm is not.

FIGURE 1. CHIP SENSOR WITH TWO WAVEGUIDES

FIGURE 2. LAYOUT OF THE TWO-WAVEGUIDE CHIP PACKAGE

All of the components are encapsulated to ensure ruggedness and optical stability. The unit is completely sealed with only the waveguide exposed to the sample. The actual size of the unit is $10 \times 28.5 \times 3.5 \text{ mm}^3$. The expanded circular area in Figure 2 shows one possible sequencing of the chemistry on the waveguide. The arrangement shown is for refractive index measurements. Variations of this design are available to accommodate other measurement techniques that are appropriate for the selected chemistries and biochemistries.

The selection of readily available, commercial LEDs for a light source provides the flexibility of using different detection mechanisms and chemistries. Table 1 presents only a brief example of the wide range of available LEDs. Similarly, the photo detectors can be selected to best match the requirements of that application. Table 2 lists some of the characteristics of the detectors typically used in this platform.

Table 1. LEDs

Material	8 Peak	Color
GaN	470 nm	Blue
GaAsP	565 nm	Green
GaAsP	585 nm	Yellow
AlGaAs	660 nm	Red
AlGaAs	880 nm	IR
GaAs	940 nm	IR

Table 2. Detector Characteristics

Light-to-Voltage Converters	High responsivity [80 mV/(mW/cm ²)] Low-output offset (<10 mV) Low power (3-V operation)
Light-to-Frequency Converters	Wide-output dynamic range (120 dB) Stable with temperature (100 ppm/°C) Low nonlinearity (0.1% full scale)
Linear Arrays	Single-supply operation (5 V) Single-clock operation (2 Hz) 400-dpi-pixel resolution

There are several advantages of the chip design in terms of size, performance, versatility, manufacturability, and cost. The chip package is significantly smaller than the five-optic sense assembly in the original FOCS® design. This reduction in size allows for a proportionate decrease in the size of the overall monitoring probe. There are two features of the chip package that result in an improved sensor performance: (a) the design utilizes a true reference arm that reduces noise in the output and (b) the assembly can be molded to tight tolerances and has fewer interfaces between optical components, resulting in a decrease in lost light or an increase in the signal. The chip design is extremely versatile. The reference arm allows for the negation of environmental factors or interfering analytes. One of the major advantages of the chip package is its manufacturability. Automated fabrication of the package, incorporation of several components into one subassembly with reproducible alignment of the optical components, greatly simplifies the assembly process for the total probe with resulting improvement in fabrication process yields. The resultant integrated package is rugged and suitable for field monitoring. The combination of improved sensor performance and manufacturability results in an overall cost reduction for the sensor.

3.0 ELECTRONICS AND SOFTWARE

The sensor chips used in the probe are photo detectors manufactured by Texas Instruments. The photo diodes convert light to frequency and can be directly interfaced to the internal timer counters in the microprocessor. The frequency outputs of the chip, which are directly proportional to the detected light level, are used to determine this relative change in the sense waveguides as compared to the reference waveguides.

The probe has a temperature sensor on board so that the response of the chip sensors can be temperature corrected if necessary. The temperature sensor has a temperature resolution of a few hundredths of a degree. The probe is calibrated over the operating temperature range 0–50°C.

The probe as well as the sensors are designed to be versatile. They can operate in water as well as vapor. This dual operation requires the microprocessor to store calibration curves for operation in both media. The use of a media sensor allows these processors to switch to the appropriate calibration curve.

The drive current for the individual sensors on board the probe can vary depending on the sensor that is used. Therefore, the drive current is programmable for each sensor. The LED in the sensor can be switched on continuously or pulsed. The power duration as well as the frequency of the LED can also be programmed. The probe has an RS-485 interface "multidropped differential drive" as well as a "talk" signal that allows the probe to be used with repeaters and directly with radio networks. The communication protocol is programmable. To facilitate use with a wide variety of controllers, the requirements of the probe are kept low. The system voltage can be from 9 and 24 volts. The microprocessor is reprogrammable, allowing for a change in the function of the

probe by replacing sensors.

4.0 PROBE

The construction of the probe can take either of two basic designs. The first form, shown in Figure 3, has two modules with a connector between them—a sensor module and a processor module. Thus, the processor module can accommodate a variety of three-chip sensor modules. The second form is simply the elimination of the connectors, allowing all of the components to be molded into a single module. In either form, the probe will have an external housing to protect the waveguides and provide some handling protection to the rest of the probe.

To maximize the types of monitoring situations that can be accommodated by the design, the probe housing has an outside diameter of 19 mm (0.75 in). The probe length is determined by selecting one of two basic forms for the application. In the single-module form, the probe is constructed to withstand submersion to 75 m (250 ft). The assemblies are molded to either a connector or a cable, depending on the requirements of the data-gathering system. When molded to a cable, the type and length of the cable is dependent on the application.

5.0 APPLICATIONS OF THE THREE-CHIP PROBE

The three-chip probe is particularly suited to applications where more than one simultaneous measurement is needed to obtain the requisite information. Possible applications include the following:

- # Measuring the release of toxic and hazardous compounds. An example of this use is the monitoring of training and testing activities at the U.S. Department of Energy's (DOE) Hazardous Spill Center (HSC) in Nevada.^a The

FIGURE 3. CONSTRUCTION OF THE THREE-CHIP PROBE

initial probe may have sensors for polar, nonpolar, and acid/base compounds to give the broadest nonspecific coverage. One or more of the three-chip sensors can be changed to give specific species information.

^a

The HSC is a research and demonstration facility available on a user-fee basis to private- and public-sector training sponsors concerned with

the safety aspects of hazardous chemicals. Though initially designed to accommodate large gas releases, the HSC can also accommodate hazardous materials training and safety-related testing of most chemicals in commercial use. The HSC is located at the DOE's Nevada Test Site near Mercury, Nevada, USA.

- # Measuring the progress that is being made in bio-remediation efforts. A probe containing hydrocarbon, oxygen, and carbon-dioxide sensors would be very practical in assessing the efficacy of hydrocarbon cleanup.
- # Measuring the presence and/or exposure to chemical warfare agents. A probe containing three sensors that would detect chemical agents in various ways (e.g., by enzyme and by direct agent detection) would give improved protection.
- # Measuring the presence of potentially harmful or toxic situations in agriculture or horticulture. The probe could have sensors for pesticides, herbicides, and fertilizers.
- # Measuring the presence of organic chlorides. Since the probe is designed to operate to a water depth of 75 m (25 ft), "sinkers" such as trichloroethylene (TCE), tetrachloroethane (TCA), and chloroform could be measured at the bottom of a well.

6.0 PRELIMINARY CHIP MEASUREMENTS

At the present time, several chip-level sensors are under development. Of these, two are amenable to the three-chip probe— hydrocarbons and atrazine. The chemistries-biochemistries used on these sensors are proprietary and will not be discussed.

The hydrocarbon sensor^{7,8} is a chip-level, single-step sensor that uses proprietary FOCS® chemistry. A change in refractive index can be directly related to the amount of hydrocarbon present. Figure 4 shows the response of this sensor to gasoline, and compares it to data taken with a nondispersive infrared (NDIR) system. The data were taken by flowing clean air and then a gasoline mixture over the sensor and into the NDIR unit. As can be seen, both sensors are reversible, but the response of the chip-level sensor was much faster: 3–5 seconds versus 40 seconds. The NDIR system response is determined by the time that it takes to fill the sample cell. In actuality, the chip sensor represents a continuous monitor while the NDIR is a batch-sampling device. The chip level returns much more quickly to base line as air is injected and the hydrocarbon concentration decreases: 10 seconds versus 40 seconds. The difference is related to the time it takes for the hydrocarbon to leave the surface of the chip as compared to the time it takes to flush the cell. The fast response and simplicity of the chip sensor makes it ideal for monitoring and detection applications.

FIGURE 4 A COMPARISON OF A CHIP SENSOR TO THE NDIR SYSTEM FOR HYDROCARBON DETECTION

The atrazine⁹ sensor is an example of a herbicide measurement device. This sensor is a solid-state, single-step irreversible competitive immunoassay that can be applied to the sense arm. The basic technology is intended to be the basis for a series of sensors for environmental monitoring and military threat-warning sensors. It will respond to vapor, aerosol, and liquid targets. The 10-ppb sensitivity shown in Figure 5 does not represent the lower-detection limit. Applying the coating to a chip produces a low-cost sensor that can be either regenerable or "throw away."

FIGURE 5. RESPONSE OF A CHIP SENSOR TO ATRAZINE

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KEYWORDS

hybrid sensor
optical waveguide sensor
optical chemical sensor
optical biochemical sensor
sensor

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ATTACHMENT D

CONFERENCE PAPER

SELF-CONTAINED, HAND-HELD, OPTICAL WAVEGUIDE
CHEMICAL SENSOR SYSTEM

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Self-contained, hand-held, optical waveguide, chemical sensor system

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ABSTRACT

A self-contained, hand-held, optical waveguide, chemical detection system has been built to detect and quantify gasses and vapors. The system uses a hybrid integrated circuit (IC) containing optical waveguides coated with sensing chemistry as the optical platform. The IC with sensing chemistry is available commercially under the name Sensor-on-a-Chip®.¹ This IC is mounted in a small, uniquely designed sample chamber where the measured analyte is identified by the sensing chemistry and biochemistry. Continuous or stop-flow sampling is possible. Sensitivities in the low parts-per-million have been attained for hydrocarbons and alcohol. Analyte coverage is only limited by the sensing chemistries and biochemistries that are available.

Keywords: hand-held sensor, optical waveguide chemical sensor, Sensor-on-a-Chip®, chemical sensors, biochemical sensor

1. INTRODUCTION

Ever-increasing needs for environmental safety in the public arena and the workplace coupled with the potential for worldwide threats from chemical and biological terrorism are the driving forces behind the growing interests in threat-specific and broad-area chemical and biological sensors. As well as providing point detection with hand-held devices, it is important to be able to provide broad-area coverage by incorporating many point detectors into a network. The work described in this paper is intended to provide a solution for producing a low-cost, hand-held or pocket-sized detection system. The system contains a computer interface for potential networking and could have added radio-frequency (rf) communications for remote locations. The heart of the detector is an optical sensing system hybridized into a commercial IC.

The use of optical waveguide, chemical, and biochemical sensor systems has been limited by size and cost. To achieve broad acceptance, these sensor systems must be small and reasonably priced. Additionally, these

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systems must be capable of detecting and quantifying single and multiple analytes, and they must possess the specificity, sensitivity, and accuracy compatible with their proposed application. A hand-held sensor system that meets these requirements has been constructed, tested, and evaluated.

Optical waveguide, flat or fiber optic, chemical²⁻⁸ sensors are the transducers in an information acquisition strategy where *in-situ*, real-time data are obtained to determine the presence and concentration of individual species, or groups of compounds, in chemical systems. Sensing indicators are attached to the surfaces of the waveguides so that preselected chemical, biochemical, and/or physical properties can be measured. Recent advances⁹⁻¹² in sensor and optical technologies have made it practical to design and build a self-contained, hand-held sensor package that can detect and quantify analytes including vapors and gasses. The analyte is identified by the sensing chemistry or biochemistry. An adaptation of this technology that will make it possible to measure aerosols and liquids is currently under development.

The two, key assemblies of the hand-held chemical detector include the hybrid IC optical waveguide sensor platform¹ and the sampling chamber. The sensor assembly uses two miniature coated waveguides (sense and reference) attached to an IC containing a single light source, two identical detectors, and the requisite optics to guide the light from the source to both the sample and reference waveguides and then onto the detectors. A fan is used to pull the analyte through the chamber. This fan is operational during the sampling mode and stops to provide a static sample when the measurement is being made.

The hand-held unit is software-controlled. This software provides the concentration of the analyte displayed in parts-per-million (ppm) and an alarm that can be set to alert the user when a desired level of concentration is reached. The instrument calibration can be verified before and after each measurement. The chamber can accept interchangeable sensor packages so that a number of analytes can be measured. The prototype instrument (dosimeter) measures $3 \times 7 \times 1$ inches and weighs 12 ounces, but it can be further miniaturized. Without power, the fan and electronics are off, but the sensor still responds to changes in the environment.

2. SENSOR

Selecting the optical sensing platform (IC sensor module) is an important consideration in composing the total sensor package. Using miniature waveguides on a hybrid, semiconductor IC is an ideal solution for designing a hand-held unit that is rugged, reliable, and small-sized. Additionally, a product that possesses these characteristics is available commercially.⁸⁻¹² The hybrid configuration is a mass producible, 20-pin, dual in-line IC package with sense and reference waveguides mounted above the top of the IC. Figure 1 is a concept drawing of the sensor showing the waveguides as hatched elements between the molded-prism, light deflectors. Figure 2 is a photograph of the sensor IC with the two waveguides prominently visible on top, and Figure 3 is a schematic showing the optical layout.

The IC uses miniature waveguides as the active elements and measures only $1\frac{1}{4} \times \frac{1}{2} \times \frac{1}{4}$ inches ($30 \times 12 \times 6$ millimeters). It consists of a centrally located, light-emitting diode (LED) that projects onto a pair of splitter prisms. These prisms direct the light into a pair of waveguides: one arm is used for sensing and the other is used for reference (or for additional sensing). The waveguides direct light toward the two prisms that couple the light respectively to a sense optical detector and a reference optical detector. The waveguides operate as "light pipes" thus providing optimum use of the sense and reference chemistries and the light budget.

The hybrid IC solves the problems associated with existing optical waveguide configurations where the entire sensor is not integrated. Therefore, the hybrid IC eliminates concerns such as optical alignment and the need for an external light source and detector. The commercial IC package is encapsulated so that only the waveguides are exposed to the analyte. A temperature sensor and a processor with memory are combined to form the electronics

module, which converts the output of the sensor module to concentration. The serial memory provides performance and calibration information about the specific sensing chemistry or biochemistry. This information can be provided for each interchangeable plug-in sensing module. The pins shown in Figure 1 attach the IC to the data gathering and -processing modules.

The insert in Figure 3 shows how the chemistries, in a solid-state configuration, can be arranged in a preselected, geometric sequence to control the reaction of the analyte with the sensor while protecting the sensor from interferences. The sensing arm is coated with chemistries that are selected to be as specific and as sensitive to the target analyte as possible. The reference chemistries are selected to have equivalent properties (*i.e.*, temperature coefficient and humidity response) as the sensing chemistries but do not interact with the analyte. It is imperative that the selected chemistries or biochemistries are not affected by the medium where the target analyte is measured. In some cases, an overcoating that acts as a membrane is sufficient to surmount any interference problems. Sense chemistries for hydrocarbons and ethanol have been incorporated into the hand-held instruments.

3. SAMPLE CHAMBER

A miniature sample chamber was designed and constructed for the hand-held instrument to be compatible with the hybrid IC. The two, key design requirements for the miniature sampling chamber were (a) the capability to bring samples to the waveguide surfaces without altering the samples and (b) the capability to prevent ambient light from reaching the chip. Various transfer mechanisms such as fans and pumps—operating continuously or intermittently—were evaluated as methods for bringing the analyte to the waveguides.

The design that was selected for the hand-held unit is an axial-flow manifold with a miniature fan operating intermittently as the air transport mechanism. The air enters the inlet manifold, passes through a foam filter into the sample chamber, flows through another foam filter, and then passes out of the sample chamber through the fan. The fan is used in the exhaust mode to minimize turbulence over the waveguides of the chip and to maintain the integrity of the sample. The foam filters are used at either end of the sample chamber to provide anti-backflow baffles when the fan is not running. The filter at the inlet of the sample chamber also serves to smooth the sample flow from the inlet manifold and attenuates some of the ambient light.

The miniature sample chamber is configured to acquire baseline and analyte vapors from the environment and presents the samples to the sensor waveguides. The chamber provides various features such as ambient light baffling, flow smoothing, and sample retention for operating the system intermittently. The layout of the sample chamber is shown in Figure 4.

The air transport mechanism was designed to move at least three volumes of air (and preferably five volumes) between samples. Each volume includes the sample chamber and upstream chambers and tubes. This is standard laboratory practice for vapor sampling and is intended to ensure that the new sample has received minimal dilution from the previous sample. The design that was selected can achieve these goals while maintaining a rapid sampling rate.

The IC can be configured to use LEDs over a wide spectral range extending from the blue to the near-infrared. Ambient light must be blocked from the sensing elements; otherwise, the photo detectors will see the ambient light thus overloading the electronics. Many measurements, especially for absorption, exist at wavelengths that are in the visible band. The total effect of direct external light could be deleterious to these measurements. In addition to blocking ambient light from entering the chamber, the ICs are coated in the inactive areas to further minimize the transmission of ambient light through the housing. Results from tests conducted to measure the transmission of ambient light showed the coating process to be effective. It is important to note, however, that the complete absence of external light is not essential since the output of the IC can be read with the LED turned

off and the ambient background subtracted or compensated in subsequent calculations.

4. ELECTRONICS AND SOFTWARE

The sensor IC module contains the electronics needed to output the raw data that are collected while the sensing and reference chemistries interact with the analyte. Additionally, the electronics and software as well as the IC configuration are matched to the individual measuring methods (*i.e.*, absorbance, refraction, fluorescence, and/or Raman effect).

In applications where sensitivity is a key consideration, each of the electronic components must have the smallest temperature response and the lowest noise available. Since both of these noise sources are additive, they can destroy the sensitivity and resolution of the instrumentation. Temperature variance is usually the main culprit. This is compensated in the electronics and by using an internal arrangement of the sense–reference waveguides. The sense–reference arrangement also provides a means for minimizing the effects of humidity. Since both sense and reference waveguides are illuminated simultaneously by the same LED, light output is regulated to give optimum performance. The detectors, however, should be matched or, at least, of the same type.

The electronics and software module operates the sensor IC. This module provides all "housekeeping" functions and power to the light source(s) and detector(s). The software must process several correctional functions to optimize detection and quantification. These functions include (a) maintaining a stable light output, (b) equalizing the response of the detectors, (c) eliminating effects of humidity, (d) canceling temperature variations, (e) abrogating the differences in response between the sense and reference waveguides, (f) accounting for volatility in the sample, and (g) obviating the contribution from the sensing medium (*i.e.*, water, air, or vapor). Many of these corrections can be accomplished partially or fully using the reference sensor. The degree to which other corrections have to be made will depend on the ultimate requirements for sensitivity. The software is written so that the output is a direct reading of the analyte concentration.

5. APPLICATIONS

Possible applications of the hand-held sensor system include (a) personnel protection, (b) home monitoring, (3) evaluating work place atmospheres, and (d) environmental protection. The types of compounds and materials that can be measured include (a) solvents, (b) polar compounds, (c) nonpolar compounds, (d) chemical agents, (e) biological agents, (f) heavy metals, (g) halogenated hydrocarbons, (h) explosives, and (i) illicit drugs.

The following list contains typical applications for personal dosimeters and point detectors:

- # Chemical and biological agents
- # Solvents
- # Polar and nonpolar compounds
- # Manufacturing and industrial materials
 - Starting materials
 - Intermediate processes
 - Final compounds
- # Workplace atmospheres
- # Drugs
 - Starting materials
 - Intermediate processes
 - Final compounds
 - Degradation products

- # Explosives
 - Starting materials
 - Intermediate processes
 - Final compounds
 - Byproducts of explosions
- # Environmental pollutants
 - Air
 - Water
 - Soil

The uses for the hand-held device could extend from point-source monitoring to personal dosimetry to field screening, perimeter protection, and broad-area protection. These devices possess optimal value if they can be used in less than ideal environments, which might include dust, rain, mud, and accidental immersion. These criteria can be achieved with pocket-sized, solid-state, optical waveguide-based, chemical and biochemical sensing systems.

6. CONCLUSIONS

The prototype hand-held sensor instrument ($3 \times 7 \times 1$ inches) is shown in Figure 5. The on-off switch can barely be seen protruding from the top of the instrument. The connectors for data output and the optional external power source as well as the sampling exhaust port can be seen at the base. The display and tactile user controls are shown on the front panel. The prototype sampling input port has been modified to allow the insertion of a sampling tube and has been moved from the front to the side of the unit since the photo was taken. Table 1 lists the features of the prototype as well as the developmental goals.

Table 1. Existing and Planned Features of the Hand-Held Sensor System

Feature	Existing	Planned
Types of measurements	Absorbance, refraction	Fluorescence, Raman
Types of samples	Gases, vapors	Liquids, aerosols
Interchangeable sensor chips	Yes—by manufacturer	Yes—by individuals
Multiple sensors	No	Yes
Storage of chip coefficients	Yes	Yes
Analytes	Hydrocarbons, alcohol	Limited only by available sensing chemistries and biochemistries
Zeroing functions	Yes	Yes
Menu selections	Yes	Yes
Sensitivity	Low parts-per-million	Parts-per-billion or lower
Accuracy	15%	5%

Feature	Existing	Planned
Precision	20%	5%
Operating temperature range	20 to 100°F	! 40 to 160°F
Operating humidity range	10 to 90%	0 to 100%
Temperature correction	Yes	Yes
Humidity correction	No	Yes
Significant digit selection	Yes	Yes
Setable moving average	Yes	Yes
Automatic LED adjustment	Yes	Yes
Sampling mode selection	Yes	Yes
RS232 output	Yes	Yes
Size: 1 sensor chip	3 × 7 × 1 inch	3 × 4 × 1 inch
Weight	12 ounces	8 ounces
Size: 3 sensor chips	Not available	3 × 6 × 1 inch or 3/4 inches in diameter × 9 inches long
Weight	Not available	10 or 12 ounces
Environmentally hardened	No	Yes
Add-on rf communication	No	Yes

There is a continuing need for small, self-contained, reliable, low-cost (design goal is under \$500 for quantity) instrumentation for *in situ*, real-time monitoring of toxic species. Based on these criteria, we have successfully designed and developed a prototype and a working model of a pocket-sized, rugged, low-cost personal chemical or point detector. The model utilizes a recently developed, optical waveguide chemical sensor. We believe that this prototype, made possible by using the hybrid IC and a unique sampling chamber, is a giant leap forward in the technology of point-sampling instruments and dosimeters. With the appropriate communications and power source, the output can be fed into monitoring grids of buildings, emergency response databases, and even Internet-based national or international monitoring grids. The device is suitable for detecting toxic species, industrial chemicals, drugs, and explosives.

7. ACKNOWLEDGMENTS

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Figure 1. Concept Drawing of IC Detector

Figure 2. Photograph of the Commercial IC Detector (Sensor-on-a-Chip®)

Figure 3. Schematic of the IC Optics and Sensing Chemistry

Figure 4. Miniature Sample Chamber

Figure 5: Prototype Hand-Held Sensor System

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