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INTERNAL REFLECTION SENSOR FOR THE CONE PENETROMETER

FINAL REPORT

FOR THE PERIOD MAY 16, 1996 TO MAY 31, 1999

Submitted by:

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DISCLAIMER

PROJECT SUMMARY

The objective of this project is to fabricate and demonstrate an internal reflection sensor (IRS) prototype for the cone penetrometer. The IRS is a sensor that responds in real-time to almost any subsurface liquid contaminants. The IRS utilizes the variations of refractive index of different liquid contaminants as a sensing scheme. All the project objectives have been successfully met during the period of the program. A prototype IRS that can be easily integrated into a cone penetrometer was designed and fabricated in the first phase of the program. A controlled field evaluation of the IRS was also conducted during the first phase and results showed that the IRS was capable of locating NAPLs in soil. In the second phase of the program, the IRS was evaluated in the field and pushed into the ground using an actual cone penetrometer system. The IRS was evaluated at known contamination sites at the Savannah River Site and a commercial site in Jacksonville, Florida. Results of the field deployment of the IRS indicated that the sensor was able to sense the location of contaminants such as tetrachloroethylene in the subsurface.

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SECTION I

INTRODUCTION

A. OBJECTIVE

The objectives of this project are to design, assemble, test, and demonstrate a prototype Internal Reflection Sensor (IRS) for the cone penetrometer. The sensor will ultimately be deployed during site characterization with the goal of providing real-time, *in situ* detection of NonAqueous Phase Liquids (NAPLs) in the subsurface. In the first phase of this program, an IRS module was designed and assembled that interface directly into a standard cone penetrometer system. The IRS module was demonstrated in the laboratory and in a controlled field test to respond in real-time to a wide variety of "free phase" NAPLs without interference from natural materials such as water and soil of various types or dissolved contaminants. The Phase II aspect of the program involved a full-scale field testing and demonstration of the IRS sensor at known contamination sites at the Savannah River Site (SRS) and in Jacksonville, Florida.

B. BACKGROUND

The presence of NAPLs at U.S. Department of Energy (DOE) sites is cause for concern because these materials pose a long-term threat to drinking water supplies. By definition, NAPLs are free phase chemicals with low water solubilities, existing in soil and groundwater as undiluted "pools." These chemical pools are major contamination sources that are depleted only very slowly by dissolution into large volumes of water. The result is widespread pollution that can continue for many years if the NAPLs are not located and removed. Locating NAPLs is a challenging task, complicated by the fact that subsurface NAPLs are not stationary. Instead, larger pools can break up into much smaller ones that migrate (mostly through cracks and fissures) to other locations.

NAPLs fall into two categories based on their densities. Dense NAPLs (DNAPLs) are denser than water and sink in an aquifer. Light NAPLs (LNAPLs) are less dense than water and float. The most common DNAPLs are chlorinated hydrocarbon solvents such as trichloroethylene (TCE), tetrachloroethylene (perchloroethylene, PCE), carbon tetrachloride, and chloroform. DNAPLs have been found in many locations such as Savannah River (TCE, PCE), Hanford (carbon tetrachloride), LLNL (TCE), and ORNL (1,1,1-trichloroethane). The most common LNAPLs are fuels such as gasoline, diesel, and heating oils. Fuel contamination is a major problem at numerous DOE sites, most notably LLNL (gasoline), INEL (20,000 gallons of Texas Regal Oil), and Savannah River (diesel fuel).

Because of the significant threat posed by NAPLs, it is important that they be located during site characterization and quickly immobilized or removed. Remote, non-intrusive techniques that "look" into the subsurface for NAPLs would be ideal for this application because intrusion can open up new pathways for NAPL migration.

Unfortunately, the non-intrusive approach is technically unfeasible. Instead, devices such as the cone penetrometer, geoprobe, or hydropunch have been developed to probe the subsurface with minimal intrusion. These devices simply push soil aside during deployment, producing a hole about 2 inches in diameter, which can be filled with grout after measurements are performed. This approach disturbs the soil far less than conventional rotary drill boring and produces no waste. Cone penetrometers have received particular attention due to their deeper profiling capabilities. A variety of sensors, most of them geophysical, have been developed for the cone penetrometer but none of them meets DOE's need for NAPL detection. Geophysical techniques such as resistivity and conductivity have been investigated most for NAPL detection, but have been shown to be ambiguous when trying to locate NAPLs. In particular, many natural soil types produce a false indication of contamination. Another limitation of these methods is the need for an uncontaminated soil reference. Usually this means that data must be collected from other onsite locations assumed to be clean and extrapolated to the actual penetration location - a time-consuming and inexact procedure at best.

DOE clearly has a need for a reliable *(better)* NAPL sensor that can be deployed in a cone penetrometer or similar subsurface delivery system for *safer*, *in situ* characterization. One important requirement of the sensor is that it detects NAPLs in real time (*faster*) without responding to water, soil, or other natural subsurface constituents. Real-time response capability is essential because data is collected "on-the-fly" at cone delivery speeds of 2cm/sec or faster. Sensors that respond slowly or require long measurement times to achieve adequate sensitivity could easily miss a thin NAPL plume. A related issue is spatial resolution - the sensor must also be able to locate NAPLs on the centimeter scale, or less. The ability to distinguish between "free phase" NAPL and dissolved contaminants is also important because regulations governing the two are different. Additional requirements are that the sensor be of compact size and low cost, meeting DOE objectives for more fieldworthy, *cheaper* characterization.

The IRS is a relatively simple optical technique well suited for the detection of NAPLs in soil and groundwater. The primary element of an IRS is a prism or similar element whose internal reflectivity changes based on the refractive index of the medium against its sensing face. Figure 1A shows a light ray being internally reflected in a prism. The condition for internal reflection is established by the refractive indexes of the prism (n_1) and the outside medium (n_2). For a light ray to be reflected it must strike the sensing face at an angle greater than the critical angle, θ_c, defined by the following equation:

$$\theta_c = \sin^{-1}(n_2/n_1)$$

Figure 1B demonstrates how internal reflection is lost when a light ray strikes the sensing face of the prism at an angle less than the critical angle. Equation 1 shows that the critical angle for internal reflection depends on the sample refractive index (n_2). This simple relationship can be used for NAPL detection as shown in Figure 2 for a fused silica prism ($n_1 = 1.4584$) and a light ray striking the sensing face at 60°. When air ($n2 = 1.0000$) is the outside medium, $\theta_c = 43°$ and the light ray is internally reflected. However, when chloroform ($n_2 = 1.4460$) contacts the sensing face, $\theta_c = 82°$. The light ray, striking the interface at a less than 82°, is no longer internally reflected and instead leaks into the chloroform – an instantaneous event easily detected as a decrease in internally reflected light.

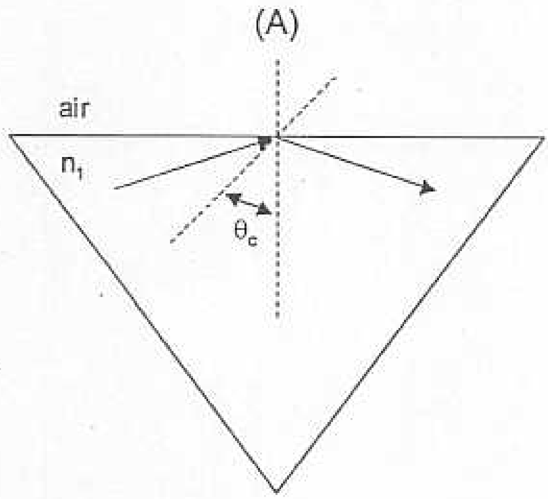


Figure 1. Paths of two light rays in a fused silica prism with air at the sensing face.

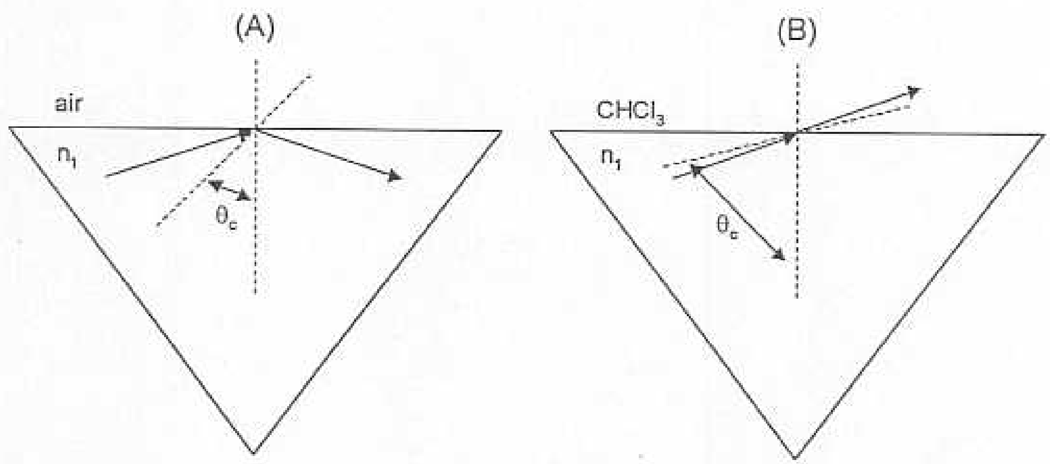


Figure 2. Paths of a light ray in a fused silica prism with (A) air, and (B) chloroform at the sensing face.

Our objective in this program is to develop a sensor for the cone penetrometer that utilizes the above principle. The sensor will continuously monitor, in real-time, the light internally reflecterd by a prism whose outer face is a "window" in the cone penetrometer.

C. SCOPE

The major technical issues addressed in the development of the IRS as a sensor for the cone penetrometer were:

- Design and fabricate a sturdy and rugged IRS prototype sensor that can be interfaced with the cone penetrometer;
- Demonstrate the effectiveness of the IRS sensor in sensing NAPLs in the laboratory and under controlled field conditions;
- Conduct a full scale field testing of the IRS sensor;
- Deliver to the DOE a prototype IRS sensor and instrument.

All of the above technical issues were addressed during the course of the program. The results of the program are described in subsequent sections.

SECTION II

RESULTS

A. PROTOTYPE IRS DESIGN AND FABRICATION

In this task, an IRS system compatible with a cone penetrometer was designed and fabricated. A schematic of the down-hole IRS sensor module is shown in Figure 3 and in appended engineering drawings. The outer housing of the IRS module is constructed of hardened steel, and all internal pieces are manufactured of stainless steel. Key sensing elements include a microlaser source, sapphire prism, and a photodiode detector. The microlaser is a low power device (<120 mA @ 5V, battery compatible) and the photodiode require no power for operation. The microlaser beam establishes the sensing area at 10 mm^2, which provides for high spacial resolution when the sensor performs measurements in the subsurface. Only four electrical conductors (two for laser power and two for detector signal) are needed for the device. Each optical element is preassembled into a mount that can then be securely inserted, yet be easily removed from the housing if replacement is necessary. The removable mount also contains a 0.25 inch diameter channel through which a standard cone penetrometer cable can pass through.

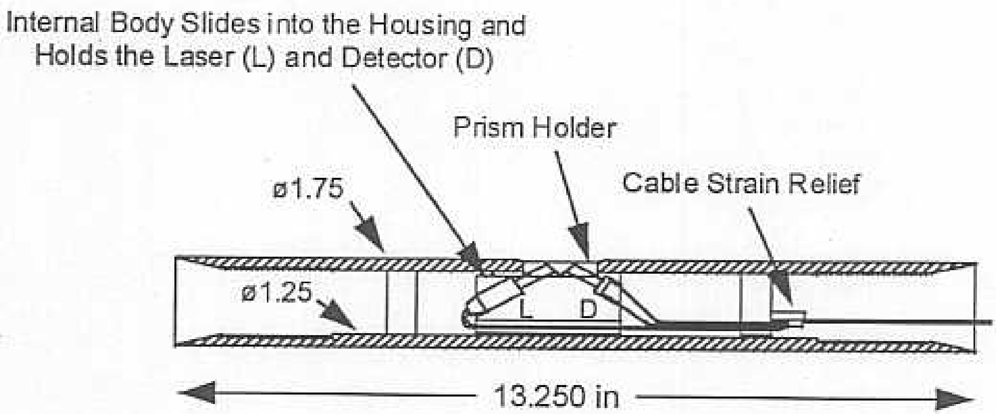


Figure 3. Schematic representation of the IRS module for the cone penetrometer.

Once the mounted optical elements are assembled and aligned in the housing, all components are fixed rigidly in place with a combination of locking screws and epoxy. There is no requirement for moving parts in the system, which renders it an extremely rugged and stable device. An end cap, equipped with a strain relief assembly for the electrical cable located at the top section of the probe, provides protection from dirt, water, etc. A hollow tube connects the end cap and the removable laser/detector unit, providing a pass-through channel for the cone penetrometer electrical cable. The overall diameter of the 13-inch long down-hole sensor module is 1.75", a standard cone penetrometer rod diameter.

Figure 4 shows a picture of the IRS module with the various components. The IRS cone section is designed to accept an internal mounting system. The inside wall of the cone is smooth and free of burrs. The thread design of the cone section allows for much of the stress to be carried by the smooth extensions into the section instead of the threads. The various taped holes allow for setscrews to hold the internal component of the IRS in place. The prism holder holds the prism at the outer cone section surface and separates the more sensitive components from the contaminated environment. The prism is epoxied into the holder. The prism holder extends down into the cone section and acts as a positive stop for the internal mount. The prism holder sits on top of the internal mount containing the laser and the detector. The internal mount holds the laser and the detector and also contains a pass through hole to allow wire that needs to go to the cone penetrometer tip to pass. The laser and detector are held in channels milled into the internal mount. The laser and detector are held at precise angles to maintain a signal for contaminants with refractive index greater than that of water. The top of the internal mount is milled flat to slide under the prism holder and a lip is left to act as a positive stop. The internal mount and the prism holder work together to align themselves when the components are put together. The laser and the detector are held in stainless steel cylinders that slide into the angled openings in the internal mount. These cylinders further protect the laser and the detector and allow for fine tuning the alignment of the components.

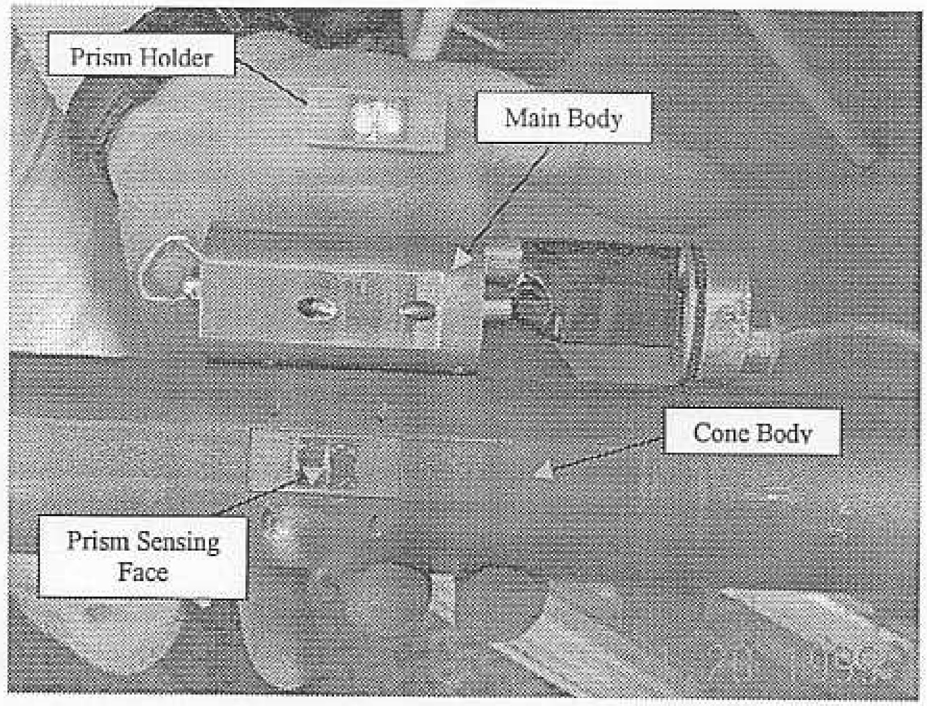


Figure 4. Picture depicting the various components of the IRS module.

The power supply for the laser and the data acquisition instrument are located uphole in the cone penetrometer truck and are connected to the IRS module via an electrical cable stringed inside the cone penetrometer rods. Data acquisition and display is done with a laptop computer via a PCMCIA data acquisition card (DAQCard-AI-16XE-50) from National Instruments. An in-house written data acquisition software was used to record the IRS response during a cpt push. The software simply records and displays the IRS detector response as a function of time.

B. LABORATORY TESTING OF THE PROTOTYPE IRS MODULE

The prototype, stand-alone IRS system was tested in our laboratory to validate its performance for NAPL characterization. To evaluate any potential interference from naturally occurring materials, a series of soil and water samples was tested first. Water samples ranged from clean distilled water to murky stream and pond waters. Soils included organic rich topsoil, sand, and several uncontaminated clays obtained from the Savannah River Site (SRS), where most of the field evaluations were conducted as discussed later in this report. The clays were collected from depths where NAPL contamination has been found and therefore represents a likely background matrix for NAPL detection at SRS. The soils were tested both wet (saturated with water) and dry.

The tests were performed by placing each material firmly in contact with the sensing prism face. Any response was measured as a decrease in laser light internally reflected by the prism; that is, a decrease in the voltage measured from the photodiode detector. Table 1 summarizes the results of the background tests, which clearly validate that the system does not respond to naturally occurring materials found in the subsurface under test conditions. For the soils, response was unaffected by moisture content; therefore, only the dry soil results are included in the table. Neither dense clays nor loose, gravelly materials posed an interference to the sensor. Because the prism is made of sapphire, none of the materials scratched or damaged the face.

Next, the response of the system was characterized with a series of 23 "pure" test samples selected to cover a wide range of refractive indices (n_D). Many of the samples were chosen in part due to their lower volatility, which ensured that they remained as a thin layer on the sensing surface while the measurement was being made. Figure 5 is a plot of percent reflectivity vs. refractive index for the test compounds included in Table 2. As expected, the internal reflectance decreases with increasing refractive index. All of the compounds, with the exception of acetone, gave strong, easily measured responses. Although the instrument can be configured to respond strongly to acetone, some natural waters have a comparable refractive index (1.36) and would produce a false positive response. We have chosen to configure the device conservatively, so that only materials of refractive index about 1.38 and higher produce strong responses. However, this refractive index cutoff is sufficiently low that virtually all the common NAPLs will produce a response. The most likely NAPLs to be encountered at DOE sites are chlorinated solvents and hydrocarbon fuels, which have relatively high refractive indexes as listed in Tables 2 and 3, and will produce strong responses.

Table 1. Water and Soil Background Test Results.

Sample	Starting mV	Final mV	ΔmV	% Reflectivity
Distilled Water	203	202	1	99.5
Tap Water	204	204	0	100
Stream Water	204	204	0	100
Pond Water	206	205	1	99.5
Topsoil Outside EIC	206	204	2	99.0
Saudi Arabian Sand	204	204	0	100
SRS Gray/Brown Clay	205	204	1	99.5
SRS Red Clay	205	204	1	99.5

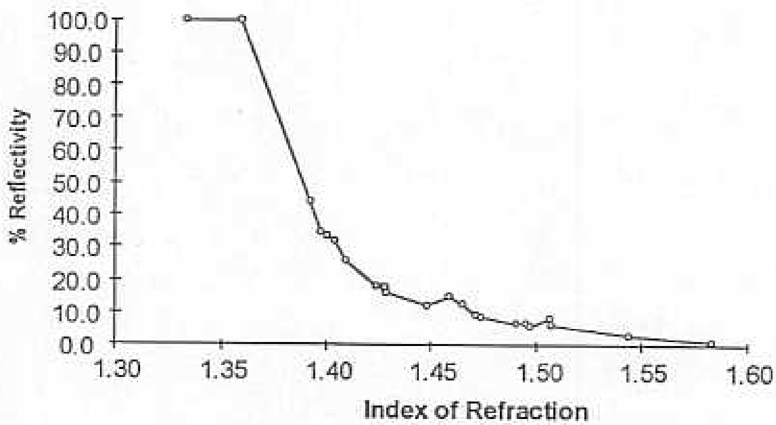


Figure 5. IRS response to various NAPLs.

In a third series of experiments, NAPLs were spiked into soil and water and the responses of the mixtures were measured with the IRS system. The water samples were prepared by adding an excess of each NAPL to tap water in a closed vial to produce the maximum aqueous concentration possible. The vial was shaken and allowed to equilibrate for several hours (to ensure saturation of the water layer) before a sample was pipetted from the water layer and placed on the IRS sensing face for measurement. Soil samples were prepared by adding 1 mL of NAPL to 2 g of topsoil collected from outside our research facility, either dry or wet, in a vial and mixing with a spatula. In some cases, it was difficult to prepare a homogeneous sample with the wet soils due to the immiscibility of the NAPLs with water.

Table 2. "Pure" NAPL Test Results.

NAPL	n_D	Starting mV	Final mV	ΔmV	% Reflectivity
Tap Water	1.333	311	311	0	100
Acetone	1.359	311	311	0	100
Isooctane	1.392	310	138	172	44.5
1-Butanol	1.397	310	108	202	34.8
Amyl Acetate	1.400	310	105	205	33.9
3-Methyl-1-Butanol	1.404	310	100	210	32.3
Decane	1.409	310	81	229	26.1
Cyclohexane	1.424	311	57	254	18.3
N,N-Dimethylformamide	1.427	311	55	256	17.7
Dimethyl Adipate	1.428	310	55	255	17.7
Ethylene Glycol	1.429	309	50	259	16.2
Cyclohexanone	1.448	311	38	273	12.2
Carbon Tetrachloride	1.459	310	47	263	15.2
α-Pinene	1.465	310	39	271	12.6
Limonene	1.471	309	29	280	9.4
Glycerol	1.474	311	27	284	8.7
Dibutylphthalate	1.490	310	21	289	6.8
ASE30 Motor Oil	1.495	310	21	289	6.8
Toluene	1.497	310	19	291	6.1
Tetrachloroethylene	1.506	310	26	284	8.4
Pyridine	1.507	310	20	290	6.5
Benzaldehyde	1.544	310	10	300	3.2
Aniline	1.583	309	4	305	1.3

The results for the saturated water samples are presented in Table 3. Clearly, the NAPLs do not produce an appreciable IRS response when dissolved at maximum concentration in water. This is not surprising as the solubilities of the NAPLs are less than 1% in water and therefore change the refractive index of water by less than 0.01 units. The IRS has been configured so that a refractive index shift of this magnitude from water goes undetected. This provides the necessary discrimination between "free phase" NAPLs and dissolved contaminants.

Table 3. Test Results for NAPLs Dissolved at Maximum Concentration in Water.

NAPL	n_D	Starting mV	Final mV	ΔmV	% Reflectivity
Trichloroethylene	1.476	210	209	1	99.5
Tetrachloroethylene	1.506	210	209	1	99.5
Toluene	1.497	211	211	0	100
Gasoline	-----	210	209	1	99.5
10W-40 Motor Oil	1.495	209	206	3	98.5

The spiked soil sample results are summarized in Table 4. The overriding trend in the data is that the response increased (% reflectivity decreased) as the refractive index of the test compound increased. This is the same trend as for the "pure" NAPLs and confirms that the sensor was responding only to the NAPL. The wet and dry soil results were comparable; the wet soil gave a slightly lower response in nearly all cases. This is presumably due to part of the sensing region being occupied by water, rather than NAPL. The largest difference was for 1-butanol. We found it especially difficult to determine if the butanol was mixed uniformly with the wet soil. The variance is accentuated by the fact that 1-butanol falls on the steep portion of the % reflectivity vs. refractive index curve (see Figure 2). Trichloroethylene, one of the most prevalent NAPLs at DOE facilities, gave a strong response in soil.

Table 4. Test Results for NAPLs Mixed With Dry and Wet Soils.

Sample	n_D	Starting mV	Final mV	ΔmV	% Reflectivity
Acetone in Dry Soil	1.359	204	201	3	98.5
Acetone in Wet Soil	1.359	204	200	4	98.0
1-Butanol in Dry Soil	1.397	203	68	135	33.5
1-Butanol in Wet Soil	1.397	203	110	93	54.2
Gasoline in Dry Soil	-----	204	29	175	14.2
Gasoline in Wet Soil	-----	204	40	166	19.4
Trichloroethylene in Dry Soil	1.476	205	18	187	8.8
Trichloroethylene in Wet Soil	1.476	206	20	186	9.7
ASE30 Motor Oil in Dry Soil	1.495	203	9	194	4.4
ASE30 Motor Oil in Wet Soil	1.495	207	14	193	6.8
Aniline in Dry Soil	1.583	203	1	202	0.5
Aniline in Wet Soil	1.583	204	2	202	1.0

C. CONTROLLED FIELD EVALUATION OF THE IRS MODULE

A controlled field evaluation of the IRS was conducted during the week of June 8, 1997 in collaboration with Applied Research Associates (ARA) at their New England Division Headquarters in South Royalton, Vermont. The two major objectives of the preliminary tests were to: (1) test the IRS durability during a real penetration and (2) evaluate the system's ability to detect and locate NAPLs in a controlled test pit. Detection entails responding to NAPLs contacting the face of the internal reflection element. A measured response indicates only the presence of NAPL and does not identify the contaminant. Location of NAPLs refers to establishing the vertical extent of contamination by following sensor response as a function of depth. As with all optical techniques deployed in a cone penetrometer, there is no provision for providing measurements at any appreciable lateral distance from the cone.

The test pit experiments were conducted first using ARA's cone penetrometer "skid" rig. The skid consisted of a cone penetrometer mounted on a trailer with lead weights added for ballast. A portable data system was set up in the back of a pickup truck parked nearby and was connected to the rig through an electrical cable. In order to maximize the depth of each push for the IRS within the constraints of a 55-gallon drum (about 3 ft deep), a short uninstrumented cone tip was configured ahead of the IRS for these tests. This resulted in the sensing element being 12 inches behind the cone tip. The four-conductor IRS cable was connected to the cone penetrometer data system cable, which had been pre-strung through the penetrometer rods. The data system supplied +4.0vdc to power the laser and 10X amplification of the detector signal. It also provided real-time readout and display of both depth and IRS response during each push.

The test pit consisted of a cemented hole in the ground slightly larger than the 55-gallon drum "sample." A forklift was used to place the drum in the pit and position the skid over the drum. Figure 6 shows the test sample prepared by Applied Research Associates. First, clean sand was placed in the drum to a depth of about 15". Then a black plastic bag containing some of the same sand wetted with weathered motor oil was placed in the drum. The oil sample was about 3-4" thick and covered most of the cross-sectional area of the drum. Approximately 5" of clean sand was then used to cover the sample. A second, smaller and thinner (about 1" thick) sample of tetrachloroethylene (perchloroethylene) saturated sand in a plastic bag was placed in the center of the drum and covered with 1-2" of a different, local sandy loam soil. A final sample of gasoline in the sandy loam (not contained in a plastic bag) was placed in the drum as a small, 1" thick patch centered in the drum (see Figure 6). The remainder of the drum was filled with moist sandy loam.

Three pushes were made into the drum. The first, slightly off-center push hooked the edge of the tetrachloroethylene bag and dragged a portion of the sample down the hole. This smeared the contamination and produced a real, but invalid response over a depth of about a foot. The other two pushes went "cleanly" through the samples, providing accurate profiles of the contamination. The results are presented in Figure 7.

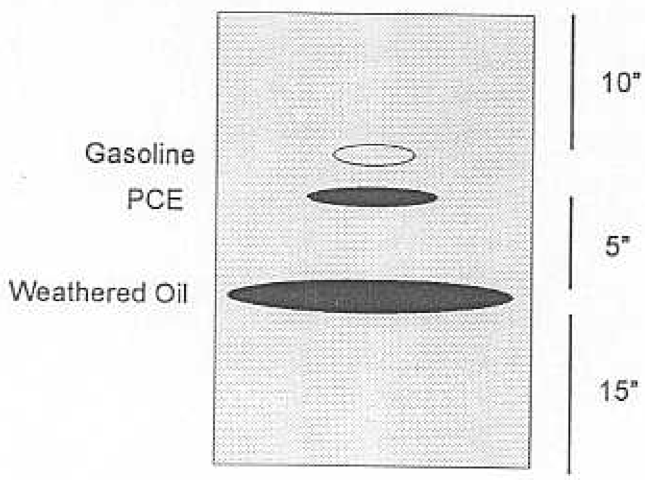


Figure 6. Cross-sectional view of NAPL test samples prepared in a 55-gallon drum.

The first of the two valid pushes (Push #2) was directed toward one edge of the drum. Therefore, it missed the two top contaminant layers, encountering just the lower weathered oil layer. The trace in Figure 7 indicates a strong IRS response to the weathered oil, beginning immediately at the depth at which the top of the material was buried (about 18" = 32" measured – 12" tip-to-sensor distance – 2" "offset" at top of hole). Note that the response persists about twice as long as expected based on the thickness of the sample placed in the drum. We attribute this to the loose packing of the freshly prepared soil sample. Loose packing may have allowed some of the contaminated sand to migrate with the cone tip and also provided less effective cleaning of the sensing surface than the more tightly packed soils of the real subsurface. Considering that motor oil is viscous and difficult to clean from glass surfaces, the results are excellent. Note also that the *IRS* gave no response to soils of different types or water contents, confirming again that the sensor does not respond falsely to natural subsurface constituents.

The second of the two valid pushes (Push #3) was positioned very close to the center of the drum. Therefore, the sensor encountered all three contaminants. Even at a much faster push rate (note the lower point density compared to the previous trace) approaching that normally used in the field, all three contaminants were easily detected. The gasoline and tetrachloroethylene layers were fully resolved from one another with only an inch or two of soil separating them. Although the layers of these contaminants were thinner, it is still evident that the response to those materials did not persist to the same extent as for the oil. This can be attributed to the lower viscosity of these compounds, allowing them to be more easily cleaned from the sensing surface during a push. It is also notable that the profile for the oil layer was the same as that observed in the previous push, demonstrating the reproducibility and reliability of the technique for locating NAPLs.

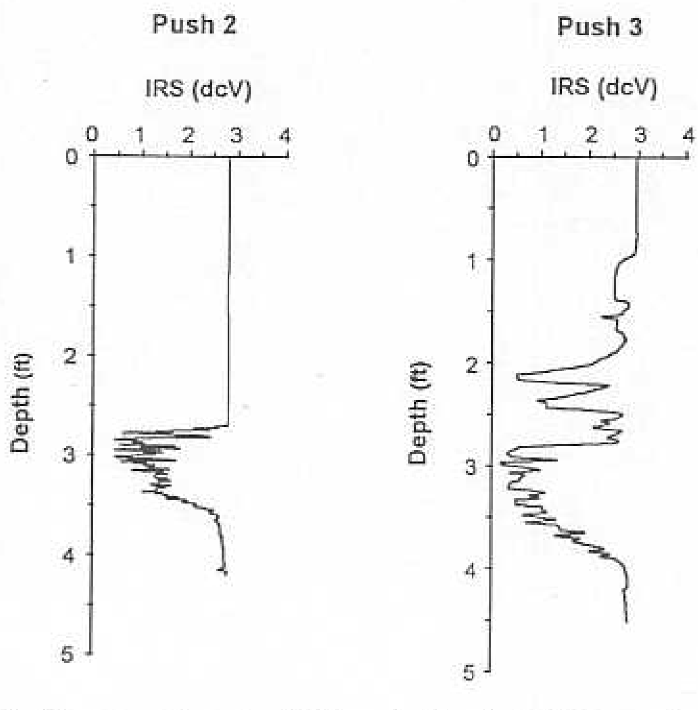


Figure 7. Cone penetrometer IRS results for the NAPL test pit sample.

As a test of sensor ruggedness, four pushes were conducted at a farm site near ARA's facility. The penetrometer skid rig was towed to the site with a tractor and set up the same as for the test pit experiments. However, the cone tip was replaced with a fully instrumented cone for these tests. The longer, instrumented cone placed the IRS sensing element 33 inches from the tip. Standard cone parameters and IRS response were monitored in real-time during the pushes. The first push reached refusal at only 19 ft; however, we were able to eclipse 30 feet in each of the other three pushes for a total deployment of over 100 ft.

The results of the four pushes are presented in Figure 8. As expected, no IRS responses were measured at this clean site. The down-hole module performed well throughout the tests, with no equipment failure even at points of high resistance and "hard" refusal. At these positions, some minor baseline shifts were observed. These may be due in part to temperature changes as frictional forces vary. We have measured the temperature coefficient of the sensor to be approximately 1mV/°C in the range 20-40°C. This temperature effect is clearly shown in Figure 8 for the first push, where the sensor baseline drifted upward as it went from the hot sun (where it had been for almost an hour) into the cool ground. Unfortunately, the cone penetrometer was not equipped with a temperature sensor to confirm this hypothesis. It is important to note, however, that the magnitude of these effects is small when compared to the magnitude of the sensor response to NAPLs.

Upon completion of the preliminary field tests, we repeated the laboratory tests with the 23 NAPLs of varying refractive index. The results shown in Figure 9 are the same as those obtained prior to the field tests (see Figure 5). This demonstrated that there was no degradation in performance of the sensor as a result of the field-testing. There was also no physical damage to the sensor that we could observe. The sensing prism face was unblemished.

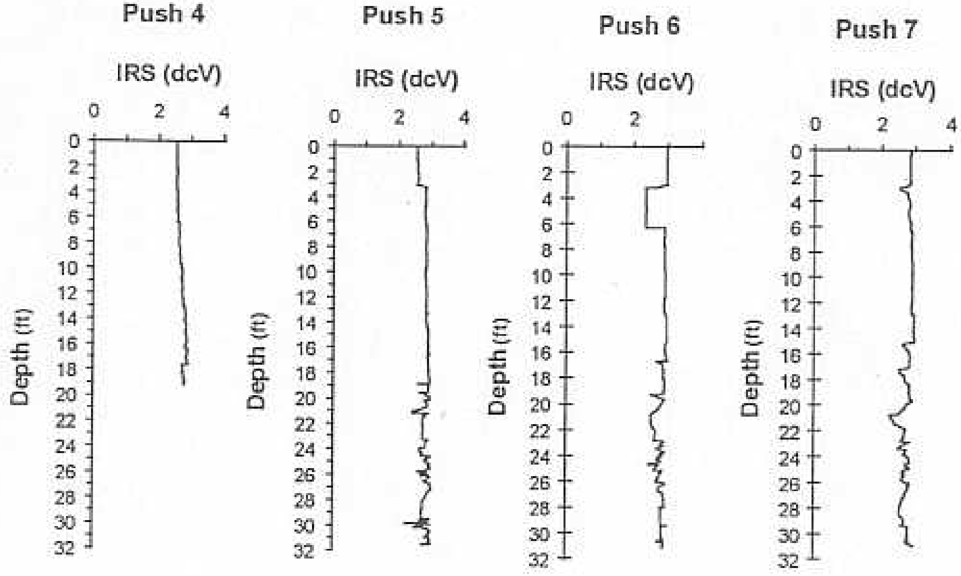


Figure 8. IRS response during cone penetrometer deployment.

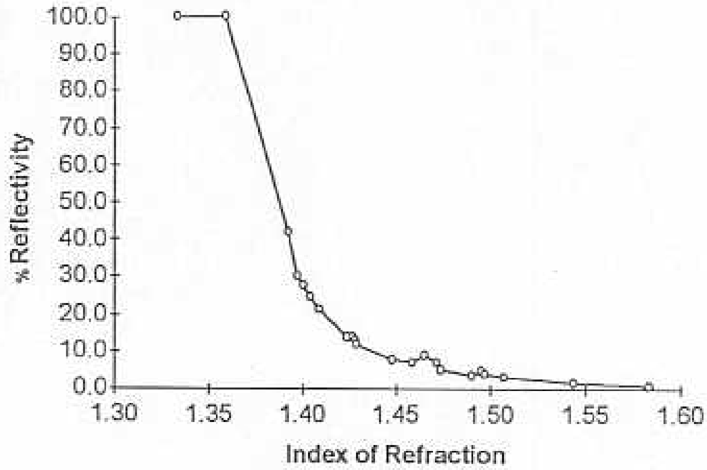


Figure 9. IRS response to NAPLs after preliminary field-testing.

D. FIELD TESTING OF THE IRS

The IRS was field-tested three times using the DOE cone penetrometer truck at the Savannah River Site. The first two tests were conducted at known contamination sites at Savannah River, and the IRS was co-deployed with a cpt Raman sensor during the deployments at SRS. The third deployment was done at a commercial site in Jacksonville, Florida.

1. SRS First Deployment

The first deployment of the IRS was conducted at SRS using the DOE cone penetrometer truck. This test was conducted the week of February 2, 1998. During this field test, a Raman sensor was also brought along and the two sensors were deployed in tandem so that the Raman sensor could be used to verify the response of the IRS. There were two pushes done with the IRS sensor during this trip. Both pushes were done at the M-Area Seepage Basin at SRS. This site is well characterized, both geological and extent of contamination. The basin has a history of dumping site of several million kilograms of waste solvents in the 1950s. These solvents, primarily tetrachloroethylene and trichloroethylene, were used in vapor degreasing operations.

During this first field test of the IRS, problems were encountered with the sensor. During the week of testing, torrential rain downpour was falling in the area for several days causing the ground to be highly saturated with water. This was a problem because water leaked into the IRS cone module during the push and damaged the IRS electronics. Thus, no useful data was obtained during the first field deployment of the IRS. A lesson learned, however, is that the sealing of the IRS needed to be improved so that water cannot get into the sensitive components of the sensor. As a result, a more liquid tight IRS sensor was designed and built after the first field-test and was used in subsequent IRS field deployments.

2. SRS Second Deployment

The second field deployment of the IRS was also conducted at the Savannah River Site the week of June 1, 1998. During the deployment, a Raman sensor was again deployed in tandem with the IRS. The Raman sensor was on a separate module that is ~3 feet above the IRS sensor and was used to help validate the response from the IRS. The deployment at Savannah River was accomplished in cooperation with Fugro Technologies, the company that operated the DOE cone penetrometer truck at that time. Three pushes were accomplished over two days. On the final push, the threads of the IRS section failed when the cone penetrometer hit a large obstruction underground. Unfortunately, the unexpected failure resulted in no further pushes with the IRS, but the failure pointed to a need to redesign the threads.

The first push was conducted at the M-area Seepage Basin at SRS. In this push, represented in Figures 10 and 11, NAPL response was detected at a depth of approximately 100 feet. The Raman sensor confirmed this response by detecting tetrachloroethylene at this depth. A response from the IRS can be seen as a rapid drop in voltage. While there are several voltage drops due to stress on the cone penetrometer, a true response will have a generally square profile. The square profile is evident in Figure 11. The response in the 100-foot range caused a rapid drop in voltage that quickly recovered as the chemical was wiped away from the sensing face of the prism. The increases within the response are due to the contaminated soil rolling over the sensing face until the cone is fully past the contaminated area.

The square profiles around the 2500-second range in Figure 10 appear to also be responses. These responses were weak and could not be confirmed by the Raman sensor. The response may have been weak because the contaminate had a refractive index close to water, causing only a slight escape of laser light out of the prism. The Raman may not have detected the contaminate because the area of contamination was small and the cone penetrometer could not pinpoint the location.

The second push at the same site, shown in Figures 12 and 13, again showed a NAPL response at approximately 100 feet. The response was also confirmed with the Raman sensor as tetrachloroethylene. The overall shape of the data profile indicates stress on the cone penetrometer. The stress results in a slight misalignment of the laser and detector, which causes the overall voltage to decrease. The overall decrease does not effect the performance of the IRS, as evidenced by the response at 100 feet. The temporary increase in voltage at approximately 1800 seconds was probably due to a temporary relief in the stress on the cone.

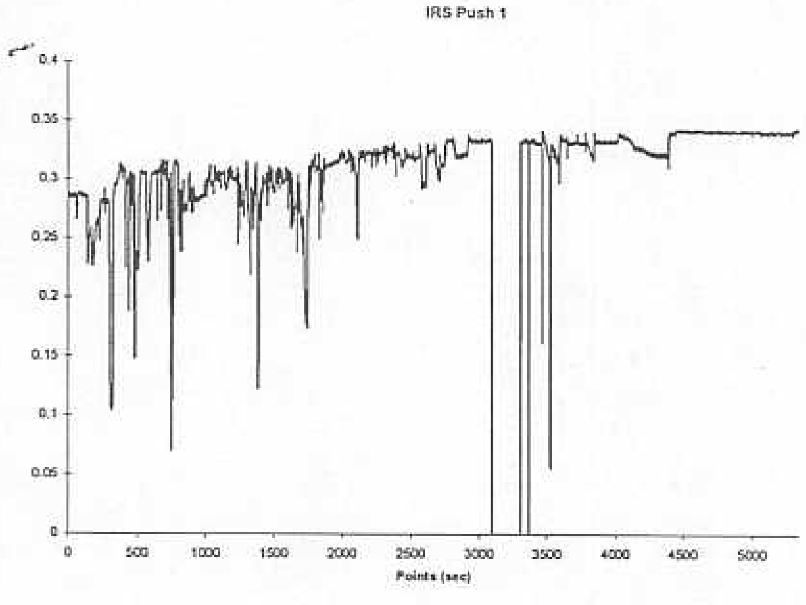


Figure 10. IRS response on the first push

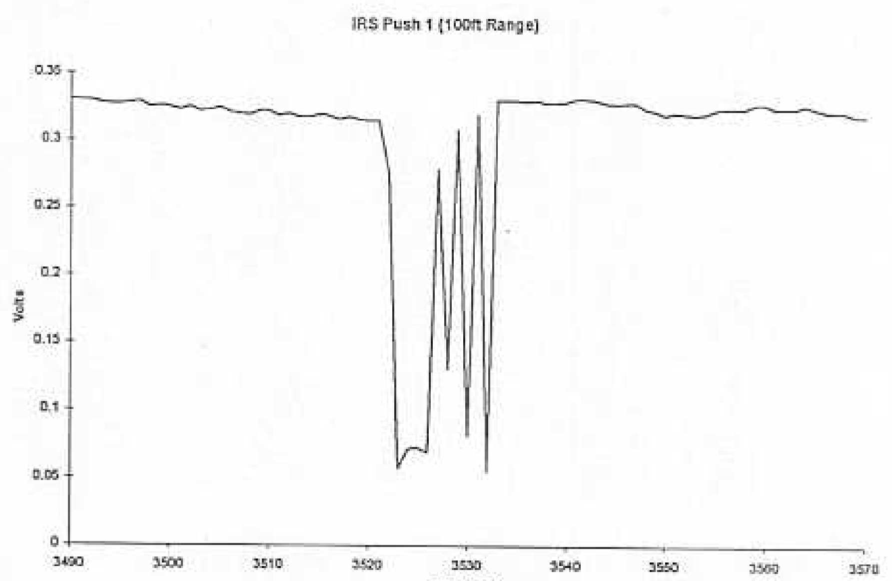


Figure 11. Response of the IRS at 100 feet in the first push.

Figure 13 shows, in detail, the response at 100 feet. The graph depicts what a good, typical response should look like. The voltage decreases as soon as the sensing face of the prism come into contact with any chemical having a higher index of refraction than water. The response will continue until the sensing face is wiped clean. The cleaning occurs simply by the sensing face passing through uncontaminated soil or a pool of uncontaminated water.

A final push was conducted at SRS in an area known as C-Area Burning/Rubble Pit. The geology, extent and type of contamination at this site were not very well known. No figures were generated from this push, however, because the cone penetrometer hit an object causing the IRS cone module to bend. The data indicated tremendous stress on the cone penetrometer, as did the data retrieved from the cone tip. The cone penetrometer probably deflected off a large object, such as a rock or buried equipment, which litters the site.

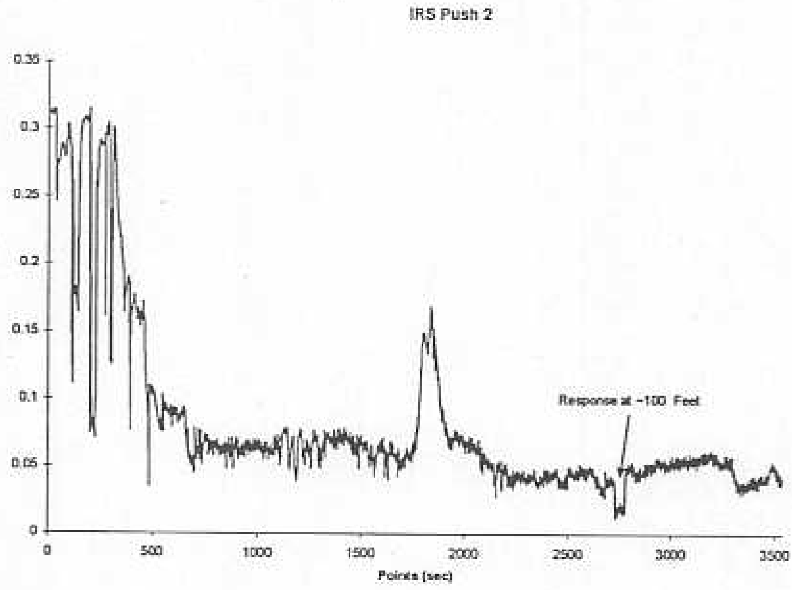


Figure 12. IRS response on the second push.

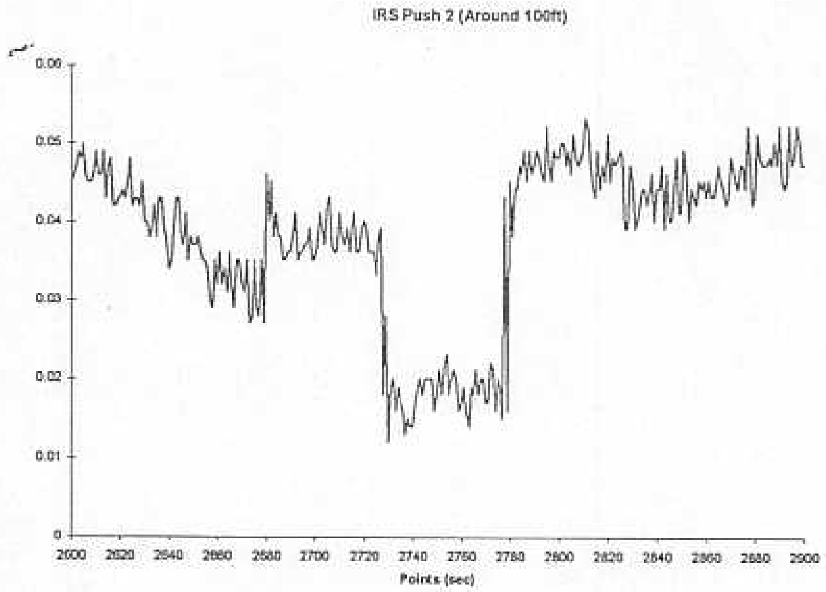


Figure 13. IRS response at 100 feet in the second push.

3. Sage Dry Cleaners Deployment

A third deployment of the IRS was done at a commercial site in Jacksonville, Florida. This site, known as Sage Dry Cleaners, was once a commercial dry cleaning site and later used as a gas station. The site is well characterized and identified to be heavily contaminated with tetrachloroethylene. The deployment at the Sage Dry Cleaners deployment was accomplished in cooperation with Applied Research Associates (ARA), the company that presently operates the DOE cone penetrometer truck. Two pushes were accomplished in one day. In the two pushes done at the site, the IRS, however, was deployed without the Raman sensor. Thus no actual validation of the IRS response was available. The Raman and other sensors were pushed separately at the site, so base knowledge exist of where the NAPL contamination can be found.

Figure 14 shows an overview of the first push at the Sage site. The laser power decreased during the push resulting in a power output too low to obtain useful data. A positive response occurred at approximately 14 feet, which is detailed in Figure 15. The response was weak, probably due to the contaminate being mixed with water. The ground morphology at the site is sandy with water throughout.

The response at 14 feet has the typical square profile associated with a response. The response at 16 feet could be either a positive response or a response to stress on the cone penetrometer. The initial drop is abrupt, like the typical response, but then decreases slowly. The response is more likely caused by stress.

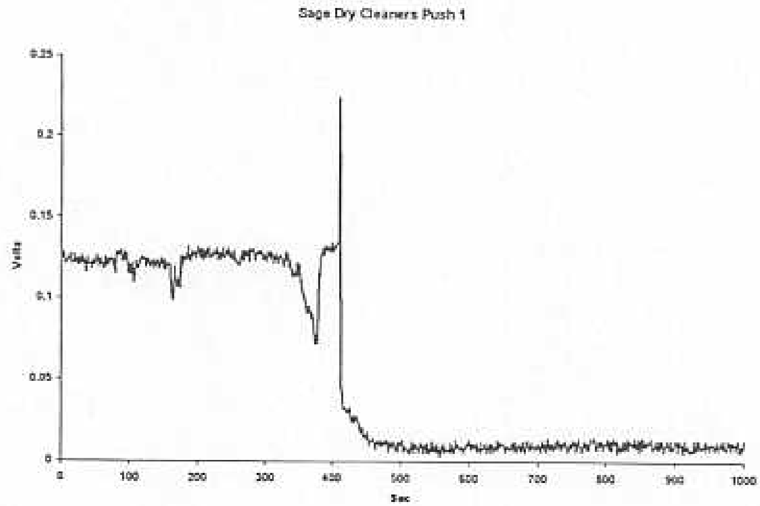


Figure 14. IRS response in the first push at Sage Dry cleaners.

Figure 15. Expanded view of the IRS response in the first push at Sage Dry Cleaners.

The second push at the Sage site is depicted in Figure 16. Unfortunately, the laser failed after approximately 22 feet. The response at 20 feet is a clear response, and corresponds to the data gathered by others at the site as to the depth of most contamination.

The response at 18 feet is clearly a response to stress on the cone penetrometer. The slow decline in signal and then the rapid recovery is typical of stress related responses. The stress responses are also detectable from within the deployment truck. The truck usually bounces and reacts to the stresses of the cone penetrometer being pushed through hard dirt or past obstructions. The stress on the cone penetrometer causes the IRS to misalign, then the IRS will realign when the stress is relieved.

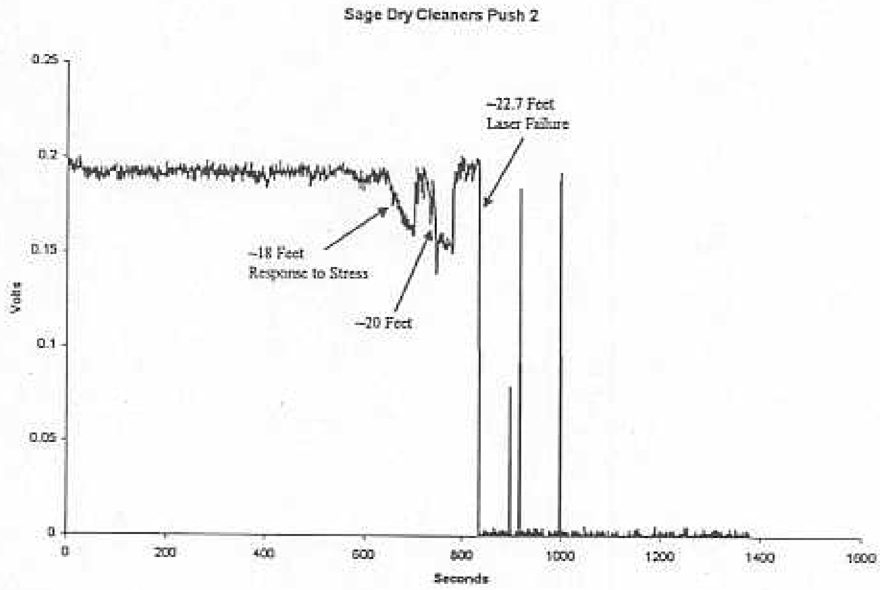


Figure 16. IRS response in the second push at Sage Dry Cleaners.

SECTION III

CONCLUSIONS AND RECOMMENDATIONS

The objective of this program is to develop a sensor that can be easily integrated into a cone penetrometer and able to sense the presence of NAPLs in real time. The sensor developed in this program is an internal reflection sensor in which NAPLs are sensed based on the differences in refractive indices of NAPLs and common subsurface constituents such as soil and water. The primary element of the sensor is a prism or similar element whose internal reflectivity changes based on the refractive index of the medium against its sensing face. As a result of this program, a new NAPL sensor with a *real- time* response that can be easily integrated with a cone penetrometer was developed. The IRS was demonstrated to successfully locate NAPLs in subsurface contamination and to provide a response in real-time. Based on the results presented in Section II, the following major conclusions can be drawn about the IRS sensor for cone penetrometers in its current state.

- The design of the IRS cone penetrometer module is easily integrated with standard cone penetrometer rods. The components and instrumentation requirements of the IRS system are simple and inexpensive making the overall cost of the IRS inexpensive.

- The IRS sensor responds strongly to a wide range of NAPLs of concern to DOE without interference from natural subsurface materials comprising soil and groundwater. The device also differentiates "free phase" NAPLs from dissolved contaminants, even when the contaminants are present at their maximum solubility limit.

- The sensor response to the presence of NAPL in real time thus allowing minimal intrusion in the normal operation of the cone penetrometer.

- Field evaluation of the IRS sensor in contaminated sites indicates that the sensor response to the presence of NAPLs such as tetrachloroethylene.

Although the results of the field evaluation of the IRS sensor are very encouraging, additional testing of the sensor and continued improvement in the sensor design are warranted. Two important issues that need to be addressed with the IRS in its current state are as follows.

- Field evaluation data presented in Section II shows that the IRS still showed considerable extraneous responses especially in the very beginning of the push. We have attributed these responses to be due to stress on the IRS module during the cone penetrometer push causing the module to flex. In its current state, the IRS probe components such as the prism, laser, detector, and probe main body, are held in place with setscrews. The setscrews were used to allow repairs to be made to the system, such as changing the laser. As the IRS becomes widely used, however, IRS components need to be held in place in a more rigid manner such as welding or brazing. In particular, the IRS probe main body and the prism holder (see Figure 4) needs to be attached permanently into the cone section for the IRS. In this manner, the whole IRS cone section can be made as a throwaway unit.

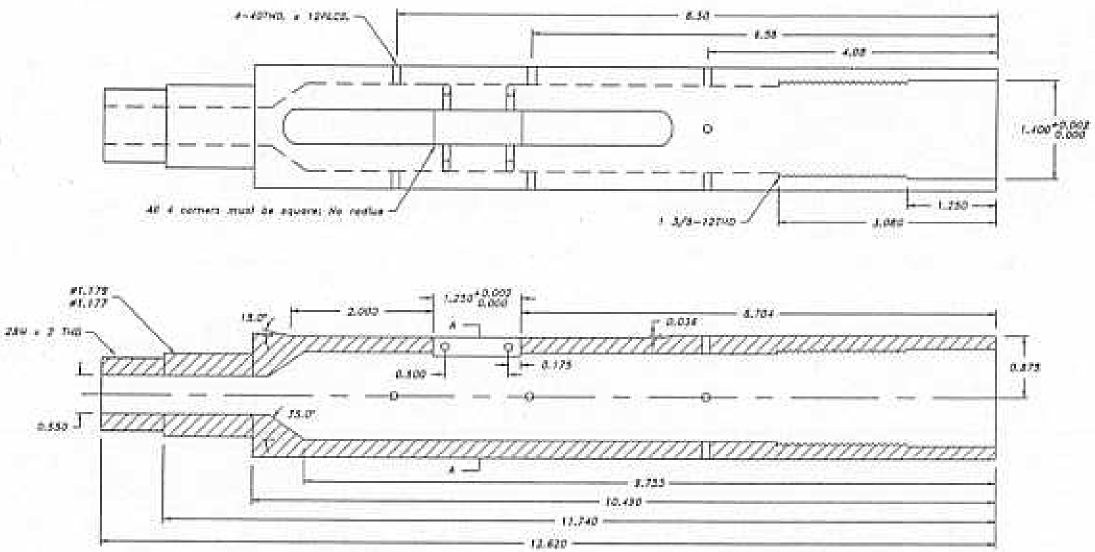
- Additional testing of the IRS with a more controlled validation of the IRS response with another cone penetrometer sensor such as the Raman or laser induced fluorescence (LIF) sensor needs to be done. This can be done by using the IRS in tandem with a Raman or LIF sensor during a cone penetrometer push and validating the response of the IRS with the Raman or LIF sensor. Although this was done in some of the cone penetrometer pushes at SRS, additional validation pushes and more controlled validation experiments are still warranted.

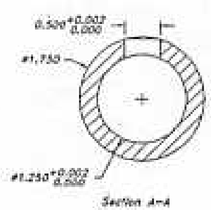
All the objectives of this project have been successfully met. A cone penetrometer sensor based on an internal reflection sensing scheme has been developed and shown to respond in real-time to the presence of NAPL. Data from field deployment of the IRS show that the IRS is capable of locating NAPLs in the subsurface. A prototype IRS sensor that easily integrates with a cone penetrometer has been built in this program.

APPENDIX

REVISIONS

REV.	DESCRIPTION	DATE	APPROVED
B	Change one end to external threads	6/23/97	
C	Remove four 4-40THD. holes Extend surface indent 2in per side	5/1/98	
D	Change External Threads to 28M x 2	10/5/98	

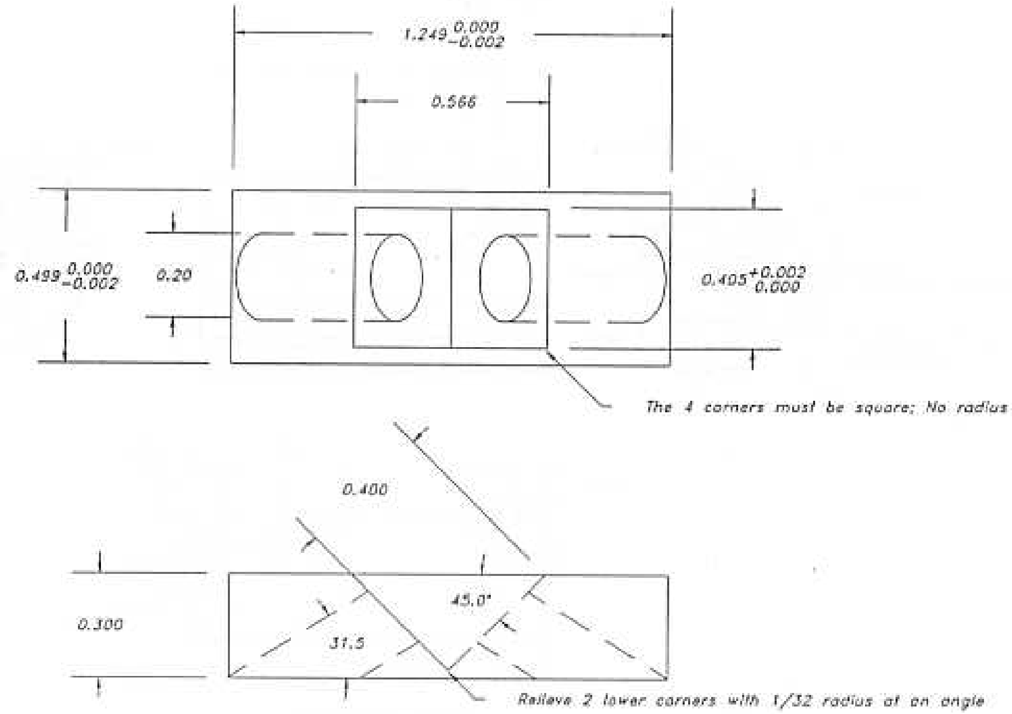




Note: Break Sharp Edges

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES. TOLERANCES ARE: x.x° = ±0.5° x.xxx = ±0.002	THE INFORMATION CONTAINED IN THIS DRAWING IS THE CONFIDENTIAL PROPERTY OF EIC CORP., AND IS NOT TO BE REPRODUCED, USED OR DISCLOSED OUTSIDE OF EIC WITHOUT PRIOR WRITTEN AUTHORIZATION FROM EIC CORP.	EIC LABORATORIES, INC. 111 Downey Street Norwood, MA 02062
MATERIAL: 12L14 Steel Case Hardened to 60RC	DRAWN: T. Johnston CHECKED: 10/5/98 APPROVED:	TITLE: Cone Section DWG. NO. 95021-7 REV. D

REVISIONS			
REV.	DESCRIPTION	DATE	APPROVED
A	Open width of triangular recess to 0.405	1/24/96	



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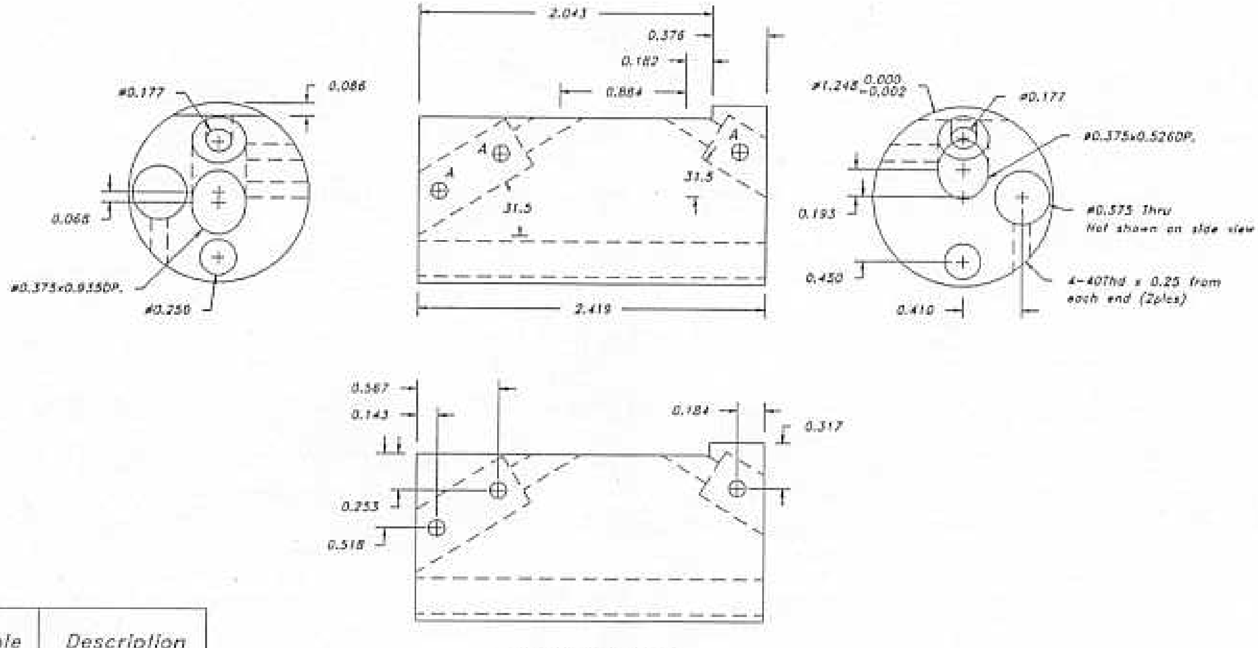
MATERIAL: 303 SS	DRAWN: T. Johnston CHECKED: APPROVED:	TITLE: Prism Holder DWG. NO. 95021-4 REV. A

REVISIONS			
REV.	DESCRIPTION	DATE	APPROVED
B	Open one Thru hole to 3/8 Add two 4-40THD holes into ø0.375 Thru hole	6/25/97	



Threaded Hole Placement

Hole	Description
A	4-40 THD.

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES. TOLERANCES ARE: X.X° = ±0.5° X.XXX = ±0.002	THE INFORMATION CONTAINED IN THIS DRAWING IS THE CONFIDENTIAL PROPERTY OF EIC CORP., AND IS NOT TO BE REPRODUCED, USED OR DISCLOSED OUTSIDE OF EIC WITHOUT PRIOR WRITTEN AUTHORIZATION FROM EIC CORP.	EIC LABORATORIES, INC. 111 Downey Street Norwood, MA 02062
MATERIAL: 303 SS Rod	DRAWN: T. Johnston CHECKED: 6/25/97 APPROVED:	TITLE: Internal CP Mount DWG. NO. 95021-5 REV. B