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Factors Affecting Thermal Infrared Images at Selected Field Sites

**J. B. Sisson
J. S. Ferguson**

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**Idaho National Engineering Laboratory
EG&G Idaho, Inc.
Idaho Falls, Idaho 83415**

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Prepared by:

J. B. Sisson, Scientific Specialist

8/3/93

Date

Reviewed by:

T. K. Honeycutt, Senior Scientist (Technical Reviewer)

8/3/93

Date

S. K. Merrill, Demonstration, Test, and Evaluation Unit Manager

7/31/93

Date

ABSTRACT

A thermal infrared (TIR) survey was conducted to locate surface ordnance in and around the Naval Ordnance Disposal Area, and a thermal anomaly was found. This report documents studies conducted to identify the position and cause of the thermal anomaly. Also included are results of a long path Fourier transform infrared survey, soil sampling activities, soil gas surveys, and buried heater studies. The results of these studies indicated that the thermal anomaly was caused by a gravel pad, which had thermal properties different than those of the surrounding soil. Results from this investigation suggest that TIR is useful for locating surface objects having a high thermal inertia compared to the surrounding terrain, but TIR is of very limited use for characterizing buried waste or other similar buried objects at the INEL.

EXECUTIVE SUMMARY

Martin Marietta conducted nighttime fly-overs of the Idaho National Engineering Laboratory (INEL) carrying a thermal infrared (TIR) imaging system to locate ordnance at the Naval Ordnance Disposal Area (NODA). During the fly-over, a thermal anomaly measuring approximately 50×100 ft was found. The anomaly was warmer than the surrounding soils and was reported as a possible contaminant plume (Martin Marietta Missile Systems 1992). Field work was conducted to verify the location of the thermal anomaly and to identify factors contributing to the anomaly.

As part of the field work, the anomaly area was surveyed using an infrared (IR) imaging system carried 50 ft above the earth's surface in a bucket truck. The temperature of the anomaly was estimated to be 3°C warmer than the surrounding soils and the boundaries found using IR imaging paralleled the boundaries of the anomaly identified by Martin Marietta.

Laboratory results from soil samples obtained from the area indicated that polychlorinated biphenyls (PCBs) may be present in the parts per billion range of concentrations. The PCBs were probably the remnants of cleanup operations following an electrical transformer leak on the site. Soil sample analysis also indicated that the volatile organic compounds (VOCs) methylene chloride and acetone could be present. Further sampling using soil gas samplers could not verify the presence of either VOC. Long path Fourier transform IR (FTIR) technology monitoring data obtained over the IR anomaly did not indicate the presence of VOCs. The source of VOCs in the soil samples was attributed to laboratory contamination. Neither PCBs nor VOCs would have caused the thermal anomaly.

Using thermal IR, an attempt was made to detect a large buried caisson, a simulated waste trench, and active 10-W heaters buried at depths ranging from 2.5 to 100 cm at a nearby site. Neither the buried caisson nor the simulated waste trench were detected from soil surface temperature data. In addition, 10-W heaters buried at 17.5 cm and deeper could not be discerned. The lack of a measurable surface response was attributed to the surface temperatures being more responsive to sun-, wind-, and dewfall-induced changes in temperature than to soil heat flux from deeper depths. The analysis shows that using IR imaging for locating buried objects at the INEL is ineffective and that the TIR anomaly could not be caused by a buried object. The thermal anomaly was concluded to be caused by the gravel pad, which had thermal properties different from the surrounding soils. TIR is useful for locating surface objects having a high thermal inertia compared to their surroundings.

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ACRONYMS

CFA	Central Facilities Area
FTIR	Fourier transform infrared
GPR	ground-penetrating radar
IR	infrared
INEL	Idaho National Engineering Laboratory
LLNL	Lawrence Livermore National Laboratory
NODA	Naval Ordnance Disposal Area
PCB	polychlorinated biphenyl
TIR	thermal infrared
USGS	U.S. Geological Survey
UTTA	U.S. Geological Survey Test Trench Area
VOC	volatile organic compound

Factors Affecting Thermal Infrared Images at Selected Field Sites

INTRODUCTION

Martin Marietta conducted fly-overs of the Idaho National Engineering Laboratory (INEL) to locate ordnance at the Naval Ordnance Disposal Area (NODA) in October 1991. During a fly-over, an area approximately 50 × 100 ft was found to have a higher nighttime temperature than the surrounding soils. Martin Marietta tentatively identified the area as a contaminant plume (Martin Marietta Missile Systems 1992). Subsequent studies were conducted to identify the position and cause of the anomaly including (a) confirmation of the anomaly, (b) a literature search of documents pertaining to NODA, (c) interviews with previous site users and investigators, (d) a search of aerial and ground-level photos of the site, (e) a Fourier transform infrared (FTIR) survey of the site to identify possible volatile organic compounds (VOCs), (f) soil sampling activities, (g) soil gas sampling activities, and (h) a thermal infrared (TIR) survey of the anomaly that was conducted 50 ft above the ground surface using a bucket truck. This report provides a description of the analysis used to conduct the studies, conclusions drawn from study results, and a historical background of the NODA site.

HISTORICAL BACKGROUND

The NODA site was used by the Navy during World War II for destruction of excess ordnance from tests of refurbished gun barrels from the Naval Gun Plant in Pocatello, Idaho. The INEL area was known as the Naval Proving Ground, Arco, Idaho. Destruction of ordnance was incomplete, and unexploded ordnance as well as fragments of ordnance were scattered over the NODA site. In 1972, the NODA site was selected as a storing and shipping area for potentially explosive laboratory chemicals because unexploded ordnance already existed on the site.

A Martin Marietta fly-over of the INEL was conducted in October 1991 to determine the applicability of using state-of-the-art TIR technology for locating ordnance on the soil surface and possible subsurface disposal sites (Martin Marietta Missile Systems 1992). The NODA site, located west of the Central Facilities Area (CFA), was included in the survey (see Figure 1).

During the fly-over, considerable amounts of ordnance and fragments of ordnance were clearly imaged using TIR technology. One image indicating ordnance and a human figure, included as a relative size reference, is shown in Figure 2. These results indicate that objects with a temperature different than the surrounding soil surface can be precisely located using TIR (Martin Marietta Missile Systems 1992).

In addition to locating ordnance and fragments of ordnance on the soil surface (see Figure 2), Martin Marietta identified an area approximately 50 × 100 ft that appeared warmer than its surroundings (see Figure 3). Uncalibrated sensors were used in the Martin Marietta fly-over; thus, accurate temperatures were not estimated from the data (Martin Marietta Missile Systems 1992).

During the late 1970s, hazardous chemicals and electrical transformers containing polychlorinated biphenyls (PCBs) were stored on the site awaiting shipment to authorized disposal facilities. A report concerning land use at the site is included in Appendix A. Since many organic compounds adsorb and emit energy in the IR region, it was hypothesized that the thermal anomaly could be the result of organic contamination. While the NODA site was being used for chemical storage and shipping, an all-weather gravel pad was laid down, and small storage sheds were erected. Aerial photos dated 1981 and 1990 show the location of the storage sheds and transformers. Since variation in thermal properties over the soil surface can produce thermal anomalies as the result of small differences in the rate of heat exchange between the soil and atmosphere, the gravel pad was also identified as a potential source of the observed thermal anomaly.

The NODA site and the TIR anomaly were visually inspected from the ground on April 15, 1992. The shape of the anomaly was found to closely follow the outline of a gravel pad within the NODA boundary. Thus, several factors that could be contributing to the TIR anomaly were identified: chemical contaminants interfering with the normal emission of the soils, the emission spectra of the gravel being different than the surrounding soils, the gravels being at a different temperature than the surrounding soils, or a combination of contaminants and gravel thermal emissions. The gravels could have a different temperature than the surrounding soils and vegetative surfaces because of variations in thermal capacity or because objects buried beneath the surface of the gravel pad interrupt the diurnal and annual heat waves generated by solar heating.

Figure 1. Map of the INEL showing the location of the NODA site.

Figure 2. Thermal IR image of the NODA site. A human figure is circled and ordnance fragments are in the boxes.

Figure 3. Thermal anomaly identified by Martin Marietta during the October 1991 fly-over. The anomaly shown darker (warmer) than the surrounding area is approximately 50 × 100 ft.

ANALYSIS

Mapping Activities

In order to compare results from the various studies conducted at NODA, a local grid system was established over the NODA gravel pad. A map of the gravel pad boundaries, the large iron gate at its entrance, and the position of shrubs on the pad, was constructed using standard plane table mapping techniques. An aerial photo dated 1981, a time period when the pad was used to store hazardous chemicals was used to digitize the boundary of the gravel pad and positions of small storage sheds and electrical transformers then on the site. The boundary of the TIR anomaly obtained by Martin Marietta was also digitized. Boundaries of the gravel pad obtained from plane table mapping, aerial photos, and the TIR image were overlaid, and the results are shown in Figure 4.

Figure 4 shows that the three methods of determining the gravel pad boundary produce nearly congruent boundaries with areas the general shape of the continental United States. Differences among the methods are attributed primarily to errors in scaling the aerial photo and TIR image onto the local coordinate system. Also, during the mapping the exact boundary of the gravel pad was not always obvious. For example, the eastern boundary has wheel ruts and scattered gravel, which makes establishing the boundary imprecise. In general these results show that the TIR anomaly identified by Martin Marietta is within the boundaries of the gravel pad.

Longpath FTIR

A long path FTIR study was carried out to detect possible VOCs in the atmosphere above the TIR anomaly, since VOCs can adsorb energy in the 8 to 12 μm range. Unlike TIR, which relies on the ability of a surface to emit IR, longpath FTIR uses an IR source emitting a known IR spectrum and a receiver capable of breaking down the IR spectrum into individual wavelengths. The IR source is placed on one side of the VOC being monitored; the receiver is placed on the other side. VOCs present in the IR beam path interact with various wavelengths of IR, and their presence can be detected and their concentrations estimated. During the first week of June 1992, an FTIR system (Nicolet Instrument Corp., Madison, Wisconsin) was set up over the gravel pad at the NODA site. The IR source was located on the east boundary (at approximately 15N,0W in the coordinate system shown in Figure 4). The IR receiver was located on the west boundary (near 5N,35W of Figure 4). The concentration of vapors emanating from an area depends largely on atmospheric dispersion. Under conditions of a stable atmosphere (usually at night or at dawn), the dispersion is less and concentrations are higher than during daylight hours; therefore, the FTIR study took place overnight in order to monitor a period with stable atmospheric conditions. A letter report summarizing test results is provided in Appendix B.

The FTIR survey over the gravel pad indicated that no detectable organic contaminants were present over the gravel pad at the time of sampling. Therefore, the TIR anomaly was not the result of organic vapors emanating from the gravel pad area.

Figure 4. Boundary of the gravel pad at the NODA site as estimated from plane table mapping (dotted line), aerial photographs (shaded region), and the airborne TIR image (heavy line). The quadrilaterals indicate sites where storage sheds (Sh), drums (Drm), a dumpster (Dmp), and electrical transformers (Tr) were positioned. The contour lines show the results of the EM-31 survey. The stars indicate positions where soil and soil gas samples were taken. Unlabeled objects are unidentified items.

Soil Sampling for Chemical Analysis

In addition to disposal of Naval ordnance, the NODA site was used to store laboratory chemicals and electrical transformers before their disposal. The locations of storage sheds, drums, and electrical transformers (Figure 4) are areas most likely to contain chemical spills and leaks. Soil sampling positions in relation to the local coordinate system laid out on the site are shown as stars in Figure 4 and are also given in Table 1. Soil samples were obtained at five locations on August 21, 1992 for analysis of organic compounds and metals.

The results of the chemical analysis conducted on soil samples are presented in Appendix C. While data in Appendix C show detectable levels of methylene chloride and acetone, a data quality review found that methylene chloride and acetone values to be within laboratory contamination levels. Following data quality review, only PCBs were found above detection limits; their estimated concentrations are summarized in Table 1. The PCB results for samples above detection limits are reported on the Pesticide Organics Data Sheets as AROCHLOR-1260.

Examination of Table 1 and Figure 4 shows two areas with low levels of PCBs where electrical transformers were stored and where a known PCB spill occurred.

In addition to the organic compounds noted in Appendix C, the soils were analyzed for metals, and the results are also in Appendix C. The chemical analysis used a hot acid digestion to extract the metals and thus included large quantities of native elements known to be present in soils and rocks throughout the West. Without knowledge of the actual mineral form, the effect of the metals on the IR adsorption spectra cannot be estimated from these data. These data have been made available to the Environmental Restoration program and are being used in developing an RI/FS for the NODA site.

Table 1. Concentration of PCBs in soil samples obtained at the NODA site.

Local coordinates ^a	Chemical compound found	Concentration ($\mu\text{g/kg}$)	Quality value ^b
09N,12W	AROCHLOR-1260	47	A
10N,23W	AROCHLOR-1260	318	A
16N,27W	AROCHLOR-1260	2251	A
25N,25W	AROCHLOR-1260	78	A

a. Sampling coordinates indicated in Figure 4.

b. Quality level A indicates values above detection limits.

Soil Gas Sampling

Soil gas was sampled passively using glass test tube samplers containing vapor collectors. The collectors consisted of two Curie-point ferromagnetic wires to which activated carbon was chemically fused. Activated carbon has a high affinity for a broad range of hydrocarbon and halogenated hydrocarbon compounds. The samplers were installed by the manufacturer's representative (NERI, Lakewood, Colorado) on September 10, 1992. The samplers were located at the same points where soil samples were obtained for chemical analysis (see Figure 4 and Table 1). The samplers were allowed to equilibrate in the soil for 25 days and were then retrieved for chemical analysis. The VOCs were analyzed using a mass spectrometer.

The laboratory results of the soil gas chemical analysis (Appendix D) indicate possible benzene and toluene contamination exist on the site at the parts per billion level. It was impossible to determine if these aromatics were the result of chemical storage activities or chemical decomposition of naturally occurring terpenes volatilizing from the surrounding sagebrush and rabbitbrush.

The FTIR uses a wide IR band that encompasses the TIR band. Thus, any contaminant that influenced the TIR results would have influenced the FTIR results. Therefore, the TIR anomaly was not the result of VOCs interacting with the natural TIR emissions.

TIR Surveys at the NODA Site

Field work at the NODA site using TIR imaging systems was conducted in two field campaigns. The first campaign was carried out to verify the presence of the TIR anomaly and to estimate the difference in temperature between the anomaly and its surroundings. A second field campaign was conducted at the NODA site, after a dual band TIR system became available. Because the temperatures estimated using TIR are functions of both thermal emissivity and temperature, the TIR anomaly could result from variations in emissivity as well as temperature. Dual band TIR systems allow estimating both temperature and emissivity.

The first campaign took place in June 1992 and consisted of operating a TIR imaging system (MIKRON Instrument Company, Inc., Wyckoff, New Jersey), sensitive in the 8 to 12- μm IR band, at a height of approximately 50 ft using a bucket truck. Oblique images were obtained over two nights. Because the scanner was capable of imaging an area only 30 $\times$ 30 ft and the gravel pad was approximately 50 $\times$ 100 ft, several images were required to form a complete image of the pad (see Figures 5 and 6). Since the images were obtained at oblique angles to the surface, they are distorted images of the gravel pad as shown in Figure 4. The actual gravel pad boundaries and other thermal features were correlated to TIR by walking around the site and identifying features visible to TIR. Temperatures of the gravel pad and soils were also taken and agreed with those obtained by TIR. Individual shrubs, pebbles, and cobbles were obvious in the TIR images obtained from the bucket truck during the nighttime. The shrubs were 2 to 3°C cooler during early nighttime because they protrude into the atmosphere and follow the air temperature closely. The cobbles and pebbles were easily seen because their thermal inertia is higher than that of the soil. The thermal inertia for gravel and sandy soil were estimated as 2.2×10^3 and $1.0 \times 10^3 \text{ J} \times \text{m}^{-2} \times \text{s}^{-1/2}$, respectively. As a result of differences in thermal inertia, the gravel warms and cools at

Figure 5. Thermal image obtained during the June 1992 field campaign. The image is a distorted view of the gravel and boundaries shown in Figure 4 because the images were obtained at an oblique angle from an elevated position 50 ft above land surface located on the south side of the anomaly.

13 14 15 16 17 18 19 20 (°C)
NODA IR Anomaly

Figure 6. Thermal image of the NODA gravel pad obtained during a period of heavy dewfall. Note that the gravel pad boundary is less distinct than in Figure 7, and note the appearance of warm areas where condensation is taking place on vegetative surfaces.

ewfall.
nce of

a slower rate than the soil (Sabins 1986). Thus, a strong thermal contrast exists between the gravel and soil during nighttime hours at the NODA site.

Thermal features visible to TIR changed significantly with local weather conditions. Figure 5 shows a distinct boundary between the gravel pad and soil and that the pad was at a nearly uniform temperature 3°C above the soil temperature at 10:29 p.m. In contrast, Figure 6, which was obtained at 2:59 a.m., shows a diffuse gravel pad boundary, and shows the gravels and soils to be at nearly the same temperature. Figure 6 also shows warmer areas appearing, which were found to be vegetative surfaces that were being wetted with dewfall. The condensation of the dew on the vegetation released heat, therefore slowing the rate of cooling and making the vegetation warmer than the gravel pad. Images obtained later (not shown here) found the gravel pad boundary to be completely obscured as dewfall progressed and wetted the gravel, soil, and vegetative surfaces. These results show TIR images to be subject to thermal driving forces that can significantly alter image interpretation such as advective cooling and condensation phenomena.

The second field campaign was carried out September 13, 14, and 15, 1992 to estimate the effect of emissivity variations on TIR images using two IR scanners. Dual-band TIR uses the ratio of energies in the 8 to 12 and 2 to 5.6- μ m bands to correct for variations in emittance in the 8 to 12- μ m region. One scanner used an InSb detector sensitive in the 2 to 5.6- μ m band and the second scanner was sensitive in the 8 to 12- μ m band (Agema Infrared Systems, Setaucus, New Jersey). Both scanners were mounted to a common pan and tilt mechanism (mounted on a bucket truck) and were operated from the ground. The dual-band survey was conducted by Lawrence Livermore National Laboratory (LLNL) personnel. A letter report containing the LLNL test results is given in Appendix E. Figure 7a shows a TIR image, and Figure 7b shows emissivity obtained from the west end of the NODA gravel pad.

The images were less dynamic than those found in the June 1992 surveys because heavy dewfall did not occur. A thermal image and its associated emissivity values are shown in Figures 7a and 7b, respectively. While considerable variation in temperature was found, the variations in emissivity appear to bear little relationship to temperature. The lack of variation in emissivity further indicates that the TIR anomaly identified by Martin Marietta is the result of the difference in temperature between the gravel and surrounding soils.

An experiment using an FTIR system (Model M2400, MIDAC, Costa Mesa, California) to estimate IR emission spectra of the soils and gravels at NODA was conducted simultaneously with the dual-band survey. The FTIR system was operated by directing the IR detector at the surface and recording the resulting spectrum. The emission spectrum obtained is shown in Figure 8 which compares the IR emission spectrum obtained for soils and gravels. The differences found in emission spectrum between soils and gravels are a result of the materials being at different temperatures.

TIR Surveys at the U.S. Geological Survey Test Trench Area

One contributing factor to the TIR anomaly, hypothesized in early discussions, was the presence of buried waste. A near ideal test location was available at the USGS Test Trench Area

(a)

(b)

Figure 7. TIR image (a) and emissivity (b) of the western portion of the NODA gravel pad and adjacent soils. The four black spots on the emissivity image (b) are thermal markers and correspond to the four white spots on the TIR image (a). Light regions on the TIR image indicate higher temperatures than the darker areas. Lighter regions on the emissivity image indicate increasing emissivity.

Figure 8. Emission spectrum from NODA gravel and soil. The differences in emission spectra are attributed to a 3°C difference in temperature between the two materials.

(UTTA) for attempting to image buried waste. At the UTTA, a large buried caisson and a simulated waste trench had been emplaced during previous studies. Figure 1 shows the locations of the UTTA relative to the INEL and Figure 9 shows the layout of the UTTA.

As part of the second field campaign, the UTTA site was imaged using dual-band IR. The dual-band TIR images were obtained by LLNL personnel and the results are presented in Appendix E. One of the images obtained is shown in Figure 10. The presence of the buried caisson is not evident in the figure.

A thermal analysis was conducted to determine why the buried caisson did not produce a measurable thermal response at the soil surface. The analysis assumed the soil surface to be subjected to periodic heating and cooling as the result of diurnal and annual temperature cycles. These periodic waves are nearly sinusoidal. When a buried object is present with thermal properties different than the soil, some of the thermal energy is reflected back to the soil surface.

The surface temperature response to the reflection can be estimated from

$$R = \frac{\delta_b - \delta}{\delta_b + \delta} e^{-2D \frac{1+i}{\delta}} = \frac{\delta_b - \delta}{\delta_b + \delta} e^{-\frac{2D}{\delta}} \sin\left(\frac{2D}{\delta}\right) \quad (1)$$

where

$$\delta = \sqrt{\frac{2\alpha}{\omega}}$$

R = reflection coefficient

δ = attenuation length (m)

δ_b = attenuation length for the buried object (m)

D = depth of the buried object (m)

α = thermal diffusivity of the respective material ($\text{m}^2 \times \text{s}^{-1}$)

ω = angular frequency of the thermal wave (rad/s).

The thermal diffusivity for the soils at the UTTA was estimated from soil temperature data obtained at the site by the U.S. Geological Survey. Table 2 presents the maximum and minimum temperatures at several depths beneath the UTTA as obtained by Pittman (1989).

Figure 9. Plan view of the UTTA in relationship to the Radioactive Waste Management Complex. Test trenches are 1.2-m diameter caissons covered with 1.2 m of soil. TIR imaging took place over the east test trench.

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Figure 10. TIR imaging over the east test trench area. Heaters were buried at depths of 25, 50, and 100 cm.

Table 2. Maximum and minimum annual temperatures estimated from soil temperature data at the UTTA (Pittman 1989).

Depth (m)	Maximum temperature (°C)	Minimum temperature (°C)	Amplitude of sine wave (°C)	Thermal diffusivity ^a (m ² × s ⁻¹)
2.1	14.8	4.5	5.15	5.2 × 10 ⁻⁷
3.8	11.8	6.9	2.45	
6.1	9.9	7.9	1.0	6.6 × 10 ⁻⁷

a. Note that the thermal diffusivity is the average value for the depth interval between temperature sensors.

For the annual thermal wave, $\omega = 2\pi/(365 \times 24 \times 3600) = 2.0 \times 10^{-7} \text{ s}^{-1}$. Using the average α in Table of $5.9 \times 10^{-7} \text{ m}^2 \times \text{s}^{-1}$ for soil, and $\alpha = 0$ for the air-filled caisson, and a caisson burial depth of 1.2 m, the attenuation lengths can be computed as 2.4 and 0 m for the soils and caisson, respectively. The reflection coefficient estimated from Equation (1) becomes -0.006. This value implies that the amplitude of the reflected annual thermal wave will be approximately 0.6% of the value seen at the surface or approximately -0.12°C. Similar calculations for the diurnal cycle result in an attenuation length of 0.13 m and a reflection coefficient of -3.0×10^{-9} for an insulator buried at 1.2 m. Because the dual- and single-band TIR systems deployed had sensitivities of 0.2 and 0.1°C, respectively, the reflected component of the annual thermal wave would be at the limits of the instruments capabilities. The small temperature response attributed to the buried caisson was overshadowed by condensation and advective processes that were highly variable over short periods of time and small spatial distances. In addition, the heat flow patterns around the caisson include lateral heat transport, which makes the thermal signature of the caisson more diffuse and difficult to observe. Imperceivable surface soil response to the presence of buried objects was indicated in previous studies (Josten 1992; Williams 1991).

For a buried object to produce the 3°C temperature anomaly observed at the NODA site, the buried caisson results require the object to be shallow, continuous, and have significantly different thermal properties than the soil. The possibility of having such a large body (i.e., 50 × 100 ft) at the site and being buried at a uniform depth seems improbable. The results of geophysical surveys (see Appendix F) further support the conclusion that no buried waste or similar objects exist beneath the TIR anomaly.

During initial discussions to list all possible factors contributing to the TIR anomaly, exothermic chemical reactions were hypothesized. Appendix A includes a list of chemical reactives known to have been on the NODA site. In order to mimic the thermal effects that exothermic chemical reactions would produce, buried heater studies were initiated.

The effects of buried waste generating heat were simulated by burying electrical heaters and monitoring the soil surface temperature. Three 10-W heaters were installed at depths of 25, 50, and 100 cm on August 26, 1992, in order to allow time for thermal effects to reach the soil surface before the dual-band survey. The three black dots shown on Figure 10 show where the heaters were buried; their effect on soil surface temperature was never apparent. The lack of response in surface temperature to the active heaters was attributed to the soil being a poor conductor of heat, to lateral temperature gradients spreading thermal effects over a large area, effectively reducing vertical heat fluxes and, to large temperature changes induced by changing weather conditions.

The thermal effects of exothermic chemical reactions taking place in the surface soil as the result of chemicals being mixed directly with soil was simulated in a shallow heater study. Heaters (10 W each) were buried 2.5, 7.5, and 17.5 cm below ground surface, and the four corners of the 1 × 1-m area were established with thermal markers. The locations of buried heaters relative to the thermal markers are shown in each image of Figure 11. Power was applied at 12:03 a.m., and the heaters were monitored over the 8 to 12-μm IR band until 7:48 a.m. Figure 11a shows the soil surface and thermal markers located at each of the four corners of the 1 × 1-m test plot before providing power to the heaters. The effects of the most shallow heater can be seen on Figure 11b 22 minutes after power was applied. The effects of the heater at the 7.5-cm depth is shown in Figure 11c 3 hours into the experiment. After observing rapid warming over the most shallow heater, its power was turned off at 3:29 a.m. to prevent obscuring thermal effects of the adjacent heater. While the thermal effects of the heaters buried at the 2.5- and 7.5-cm depths were observed, the effect of the heater buried at 17.5 cm was not apparent over the 8-hour period of this test. Figure 11d shows the temperature of the soil surface 6 hours into the experiment. The thermal plume over the heater at the 7.5-cm depth did not enlarge after that time. Without the thermal markers on the test plot, the heater buried at 7.5 cm would have been difficult to discern from its continually changing surroundings. The dramatic differences seen between Figures 11b and 11c were the result of frost forming on the vegetative surfaces of grasses and forbs surrounding the test plot area. The formation of frost released the latent heat vaporization and fusion causing those surfaces to warm or at least not cool as rapidly as their surroundings.

A thermal response at the soil surface was expected from the heater buried at 17.5 cm. A crude estimate of the expected response was computed from the analytical solution of a buried point source:

$$T = \frac{P}{4\pi\alpha\rho C} \operatorname{erfc}\left(\frac{r}{\sqrt{4\alpha t}}\right) \quad (2)$$

where

T = temperature change due to operation of heater (°C)

P = power applied to heater (W)

α = thermal diffusivity of the soil ($\text{m}^2 \times \text{s}^{-1}$)

Figure 11. Thermal images obtained over shallow heaters (a) before applying power to heaters, (b) 22 minutes following powering the heaters, (c) 3 hours following power on, and (d) 6 hours following application of power. Ringed areas at each corner of the quadrilateral are effects of the four thermal markers. Effects of heaters buried at 2.5 cm (located between the two forward markers) and 7.5 cm (located between back markers) are readily visible. The effects of the heater buried at 17.5 cm (center of the four markers) is not visible.

t = time (s)

r = distance from heater (m)

ρ = apparent density of soil ($\text{kg} \times \text{m}^{-3}$)

C = thermal heat capacity of soil ($\text{J} \times \text{kg}^{-1} \times ^\circ\text{C}^{-1}$)

$\text{erfc}()$ = complimentary error function.

Evaluating Equation (2) with $\alpha = 5.9 \times 10^{-7} \text{ m}^2 \times \text{s}^{-1}$, $r = 0.175 \text{ m}$, $P = 10 \text{ W}$, and $t = 1, 2, 3, 4,$ and 8 hours result in predicted temperature changes of $0.27, 1.25, 1.82, 2.30,$ and 3.82°C for each time, respectively. The temperature changes are crude estimates because Equation (2) assumes that the heater is buried deep enough that soil surface effects can be ignored. While the estimated temperature changes are crude, the changes are within the detection limits of the TIR imaging system, particularly following 2 hours of heater operation. These results emphasized the importance of local weather conditions to the interpretation of TIR images.

The presence of the shallow heaters was observed throughout the period of frost formation at the UTTA, in contrast the TIR anomaly at the NODA site disappeared during the period of dewfall. If the TIR anomaly had been the result of exothermal chemical reactions heating the soil, the anomaly would have been observable throughout condensation or freezing periods. Therefore, the TIR anomaly was not the result of chemical reactions and the most probable cause of the TIR anomaly was the result of differences in thermal inertia between the gravels and surrounding soils.

CONCLUSIONS

The TIR anomaly reported by Martin Marietta was the result of a gravel pad being approximately 3°C warmer than the surrounding soils at the time of the fly-over in October 1991. As a result of high thermal inertia, gravels stay warmer than surrounding soils during early nighttime hours, which makes the gravels visible to TIR imaging systems during the night. The possibility of contaminants interacting with the IR band and contributing to the TIR anomaly was rejected because of the low contaminant concentrations and irregular distribution over the gravel pad. The possibility of a buried thermal source causing the anomaly was also ruled out because, in sharp contrast to the behavior of simulated exothermic chemical reactions, the anomaly disappeared when dew condensed on its surface.

TIR should not be used for locating buried waste at the INEL. Heat transfer analyses predicted that only minuscule effects would be induced by objects buried more than a few centimeters below ground surface. The predictions of the heat transfer analyses are well supported by field work in which thermal effects of a caisson (1.8 m in diameter) buried 1.2 m deep were not observable at the soil surface using TIR imaging systems. The theory was further supported by an auxiliary experiment where the thermal effects of 10-W heaters installed at depths of 17.5 cm and deeper could not be detected at the soil surface over the 8-hour period of the test.

The TIR imaging systems did respond rapidly and accurately to the formation of dew and frost. TIR imaging was successfully used to monitor condensation and freezing phenomena where latent heat transfer took place at the ground surface. It was also successfully deployed to locate surface ordnance at the INEL. TIR is useful for locating surface objects having a high thermal inertia compared to their surroundings.

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Appendix A

A Short History of the Naval Ordnance Disposal Area

Appendix A

A Short History of the Naval Ordnance Disposal Area

The contents of this Appendix is from an unpublished report by R. C. Green. In the days when the area was used, there was no legal or environmental requirement to establish massive documentation. The only requirements were that management had to approve every operation, the job had to be performed safely, personal ingress to the area had to be controlled with road barriers and a single strand fence, and range fire potential had to be controlled through the use of firebreaks. When explosives were used, they had to be controlled within the specifications established by AEC/ERDA/DOE. Towards the latter part of the NODA use cycle, some documentation was written, which should be found within the Waste Management Data Base.

The Naval Ordnance Disposal Area (NODA) is a candidate foreclosure under 40 CFR 265, "Interim Status Standards for Owners and Operators of Treatment, Storage, and Disposal Facilities." Due to the potential human health and environmental impacts associated with both the site characterization and waste removal aspects of the effort, a hazard class determination, safety analysis, and detailed environmental documentation will be necessary. For this to be done, a chemical/hazardous material source term must be developed based upon past waste handling activities that have occurred at this site. This document provides this source term.

Prior to 1949, the landmass known as the Idaho National Engineering Laboratory was owned by the U.S. Navy and was known as the Naval Proving Ground (NPG), Arco, Idaho. The NPG provided a large area for research and testing of weapons for use of Allied forces during World War II. Naval guns, which had been refurbished at the Naval Gun Plant in Pocatello, Idaho, were transported to the NPG and tested for acceptance by the fleet. U.S. Army Air Corp aircraft from the Pocatello Army Air Base were flown to the NPG, and various types of ordnance were dropped on two large circular bombing grids, one located near Argonne National Laboratory West, and the other west of the Radioactive Waste Management Complex. Also, large explosive ordnance shots were fired east of what is now the Naval Reactor Facilities. These shots involved standard Army and Navy concrete, earth-covered (igloo) magazines containing millions of pound of explosive ordnance. The NPG also had a number of gun firing ranges, one of which was approximately 29 miles long. This range began at the Central Facilities Area (CFA) and ran north east towards Test Area North. This range could accommodate naval guns ranging from 3 to 16 inches in diameter.

NODA is what we believe to be an old Naval disposal site located north-northwest of CFA and nine-tenths of a mile north of the PTI small arms/automatic weapons firing range.

The date was approximately 1967. I was working at the Test Reactor Area as a health physics technician. I had gone out to a dumpster near the MTR reactor to survey it so its waste could be shipped to the CFA landfill. The radiation survey indicated nothing out of the ordinary, but for some unexplained reason, I looked inside and questioned its contents. The dumpster was not full so I got inside and started to forage about. I moved some heavy paper and discovered 200 pounds of oxalic acid contained in four paper bags. This was being sent to the CFA landfill. I immediately notified management and voiced my concern, stating this was a bad practice and

that we needed an interim chemicals storage area for this kind of material so that we could have time to set up an appropriate disposal operation. We removed the oxalic acid and placed it in a temporary storage area.

The oxalic acid incident led to two immediate actions. The first was a management request to inspect the plant to see if there were other chemical problems that needed attention, and the other was to see if I could find an area near CFA, CPP, and TRA that we could use as an interim storage area for chemicals found. I began the second task first. I talked to a number of the old timers asking specifically if they knew of an area used by the Navy as a disposal area. Two of them were aware that the Navy had set an area apart specifically for disposal but they couldn't tell me where although one thought it was between CF and TRA, perhaps a little west. They also told me about the bomb craters east of the NRF but I was fully aware of them due to my previous surveys. I drove west of CFA to the old AEC guard firing range and searched for some dirt roads that may lead north towards TRA. This area would be ideal because it was near most of the main facilities so we could set up roads from CFA, CPP, and TRA. In this way, if we found something really reactive, we could get off Portland Avenue quickly. It didn't take long to find a dirt road leading north from the firing range. I drove nine-tenths of a mile north to an area I had never seen before and stopped. It wouldn't have aroused the curiosity of most individuals but for me it was a treasure trove. Looking eastward a few yards I could see some depressions in the soil which I recognized as small bomb craters, much smaller than those found west of NRF. I got out of my vehicle and walked to them. It is easy to see why most people would never have thought much about them even if they had been told about their location. They were small craters, approximately 5 to 10 feet in diameter, scattered amongst the sagebrush. The crater diameters are similar to those made by large, general-purpose bombs, but the depths are much too shallow. At the time, this didn't appear too important. What a mistake. I continued to look about and realized that this was a major find; the Navy really had some type of operation going on here. This site was different than the others I had found. I came upon a mound of soil about 6 feet high, which now that I knew its location I could have spotted from the road. I could tell it was manmade but I didn't know what it was. I was digging around on the top of it when all of a sudden the soil broke through and exposed a hole about a foot across. I raced to my vehicle to grab a flashlight and returned. My light then entered a chamber which had remained hidden for probably the last 20 years. The chamber was somewhat circular and steel lined. The steel was somewhat irregularly shaped as if a caterpillar tractor had been used to push in the walls and mound it with soil. I continued to search the area observing a number of bomb craters with many chunks of high explosive spread around. Smokeless powder cans and innumerable pieces of metal fragments were spread all over the area. Close inspection indicated parts of artillery shells, bombs, and mines. There were the components of many different explosive trains from many different kinds of weapons: initiators, detonators, fuses, gains, boosters, and parts of main charges. Milligram samples of the various explosives were taken and submitted for analysis, yielding the following compositions: TNT, RDX, ammonium nitrate, picric acid, and aluminum. I conducted field tests using the analytical methods described in the U.S. Army Technical Manual and found TNT, composition B, tritonal, and amatol. The TNT was red and it did burn when ignited. I have since conducted literature searches and found out that solid TNT produces trinitrobenzoic acid upon exposure to light. It is red and more sensitive to impact than TNT.

I informed management that I had found a potential site for the interim storage of hazardous chemicals found within our plant and that it could accommodate the disposal of explosive chemicals if the need arose. I would now start a chemicals safety inspection of TRA if they would authorize me to do so. They said they wanted to inspect my possible storage site prior to moving any chemicals. I then gave a number of managers a tour of the site I had found. They did not give me permission to use this site at this time because they really didn't believe we had a significant chemicals problem. They wanted me to go ahead with the chemicals inspection within the plant and after determining if we had a problem they would likely give permission to extract the chemicals from the plant and transport them to this site for interim storage. I then gave TRA an inspection. I suspected we had a problem with chemicals but no one had ever scoped it out before. Every time I had walked through the chemistry labs it was obvious that chemicals had been collected for years and never been disposed. I found many problems and learned quickly how important it is to offer solutions to managers when you inform them of a problem. I also learned how fast you can alienate some chemists when you invade their domain and claim a risk that they are not aware of. I realized quickly that my saying that a particular chemical is extremely hazardous doesn't count for much; one must prove through the literature that it is dangerous and that it provides an unacceptable risk to personnel and plant. It was this attitude that drove me to conduct massive literature searches and to collect extensive accident histories. I knew that if I discovered a significant chemical risk I had better be prepared to be skewered by some professional in the field until I could prove it was a risk. I gave my curiosity full reign and went for it.

The inspection of TRA provided great insight into the waste management practices of the day. Chemicals were either being collected in large amounts or were being flushed down the drains or toilets in relatively small amounts. On occasion, chemicals would be placed in dumpsters and sent to the landfill or excessed to the excess warehouse. All in all, the greater proportion of old out-of-date chemicals or chemical wastes were being stockpiled within the plant. The reason for this apparently being the wisdom shown by the chemists not to disperse chemicals to the environment. These professionals generally knew enough about chemical effects that dumping these agents to the environment would or should be unacceptable. The problem was that there was no waste management program in place to accommodate nonradiological chemicals or waste forms so the materials just accumulated within the facilities.

The TRA inspection was a reconnaissance blitzkrieg, this being partially due to some managers thinking I was making mountains out of molehills. I approached it from a systems standpoint based upon an idea I had hatched up for a hazardous chemicals class I had previously given. This idea being that any chemical found within an AEC operation could be classed within one of the categories of the following hazardous materials use cycle: administration, requisition, receiving, transportation, storage, process use and handling, disposal, and emergency response. This concept enabled me to see a chemical as a component of a process that would change with time. For instance, any chemical brought onsite had to have had a program, been requisitioned, received, transported, stored, and used. If I were to dig into the documentation supporting these programs I would have a clue as to what may still exist even though the program is now dead. Needless to say I was accused of sticking my nose into areas that were none of my business. The more I was confronted the harder I looked. When the Division Manager told me to forget it, the deeper I looked. When I finally finished the TRA inspection, I had a very long list of problems. I approached my management, they reviewed it and claimed it was not a significant problem, at

which time I demanded they come see for themselves. These observations combined with my literature searches began to turn the tide in my favor. The most convincing part of my literature searches that lent the greatest credibility to my efforts were the accident histories. Management finally decided to allow the use of the Naval area I had found for interim storage of chemicals only. I knew I needed an area for disposal also but couldn't convince anyone at this time. I began moving chemicals out of TRA.

During the TRA inspection I had found almost every element of the Periodic Table and most of the inorganic and organic compounds known to mankind. Many of these were still valuable but did not have a use or program that required them. Chemists consider their chemicals their children and taking one away is like taking their firstborn. One could also see that chemicals had been ordered in bulk quantities because they were probably cheaper. I decided to concentrate upon the chemicals that posed a severe threat to health and safety. The chemists and management were more easily convinced of the risks when things "looked bad." So that was where I concentrated my attacks.

The kinds of problems I found can be categorically described as follows:

- Unlabeled "orphaned" containers
- Products of chemical reactions that were uncertain
- Oxidant/reductant admixtures
- Chemically contaminated reagents
- Corroded chemical containers
- Chemicals without ownership
- Extremely toxic materials
- Out-of-date materials
- Extremely flammable materials
- High explosives
- Self reactives/reactives
- Compressed gas cylinder problems: leakers, unlabeled
- Pyrophoric materials
- Chemicals that change their properties with time
- Carcinogens, mutagens, teratogens

- Incompatible storage
- Strong reducing agents
- Air and water reactives
- Peroxide formers.

The chemicals were packaged in vermiculite-filled cardboard boxes and transported to the Naval site and placed in interim storage. There were times I'd have to chemically decontaminate the container prior to packaging it, and on a few occasions, I'd have to desensitize the chemical prior to handling it. Also, some materials required remote extraction because of their sensitivity.

As the materials accumulated at the Naval area, a number of problems became apparent. Chemical incompatibilities were a severe problem as was protection of the chemicals from the elements. Also, it was obvious we could never qualify some materials under the Department of Transportation regulations for transport to a legal repository, if we could even find one to accept the materials. Examples of these chemicals were the explosives, self reactives, oxidant/reductant admixtures, and pyrophorics. I then went to management and asked if it was possible to set up a disposal area adjacent to where the chemicals were stored that could be used to react those materials that could not be DOT qualified or for which no disposal contractor existed. A couple of managers then went with me to the Naval site to evaluate my proposal. I explained to them we had to get some funding to establish a firebreak system and housing for the chemicals. They agreed to evaluate my requests and see if they could find the funding. In the meantime, I begged and borrowed support to prepare for the time I could start the operation. I remember strongly that no manager wanted this operation. Waste management stated categorically it was outside their mission. I, being a member of the Safety Division, was placed in a very severe position, especially with regards to the conflict of interest issues. Safety personnel review the work of others, they do not perform work. Safety Division did not have funding for this type of operation.

About this time, the word got around to the other facilities of the kinds of chemical problems we had found at TRA. Managers from CPP, CFA, NRF, PBF, ANL, and TAN started calling for similar inspections. The problems were beginning to be recognized. These managers then started to fund me for supporting their disposal requests. Now, realize that I still didn't have permission to set up a disposal area. All I could do was remove the hazards from the plants and place materials into interim storage. I continued to plead my case and continued to take many managers on tour. Funding still wasn't available but it was becoming apparent whose "shop" it was going to end up in: Safety Division and Facilities management. A deal was struck and permission was given to create a 50-foot-wide firebreak around the estimated 5-acre area and a bisection firebreak that would separate the storage area from the disposal area. These firebreaks were made and a single-strand fence was erected and two gates emplaced, one on the south and one on the north. I walked alongside the road grader and watched for ordnance and as it came off the blade. What we did then would never be allowed now. We extracted parts of bombs, including fuses, gains and boosters, and high explosives; parts of torpedoes, mines, and artillery shells.

Once the firebreaks, the single-strand fence, and the gates were established, we were given permission to dispose of dangerously reactive or explosive materials. The area was now given the name Hazardous Materials Disposal Area (HMDA). I stated that I would try to limit the materials selected for explosive disposal to only CHNO systems. These chemicals would, upon detonation, yield primarily carbon dioxide and water vapor. And with the exception of only a few materials, nickel carbonyl for example, I never violated this self-imposed constraint.

With permission given to start disposal operations I decided to begin with the explosive ordnance in the area. It was obvious this was a major problem based upon casual observations of fragmentation, smokeless powder cans, bomb casings, shells, fuses, and various kinds of explosives, sometimes in twenty-five-pound chunks. One of the first shots was a ten-pound TNT equivalent charge placed into the opening of the mounded chamber I had previously uncovered. Its detonation was a shock of major impact; the mound basically disappeared and the steel walls of the chamber blossomed like a flower to a fifteen foot diameter. Ten pounds of TNT could not do this. Sweeps of the area were made and explosives, fuses, and subcomponents of various kinds of ordnance were found and disposed of. Sometimes, when using standard explosive disposal techniques such as counter charging with high explosive, the shells would not detonate. Their explosive contents and fuses would be dispersed. I took explosive samples and had them analyzed at the CPP; the explosive was real but would not support a detonation wave. I then began using an excessive counter charge for the shells but the problem still existed. Calls were then made to Los Alamos to discuss the problem with recognized explosives technologists who confirmed by suspicions that the shells had been desensitized through some mechanism. I had considered a dead pressing phenomenon associated with the original Navy operations. It appeared that the Navy had detonated large numbers of palletized artillery shells. It is not inconceivable that some of the shells had been deadpressed by the shocks. Los Alamos stated that there were a number of possible desensitization mechanisms and that it would be difficult to precisely determine which one may have accounted for the problem.

I continued to conduct Explosive Ordnance Disposal operations, in some cases having to develop new techniques to dispose of explosives that were insensitive. One technique that worked well was a thermite cookoff process, this involving the use of aluminum/ferric oxide admixture in contact with the piece of ordnance, which could be remotely fired to heat the ordnance to its cookoff temperature or to just liquify the high explosive, which then would produce some offgassing, which changed its density. A counter charge would then be fired to initiate the piece of ordnance. I consumed 2,200 pounds of thermite using this disposal technique. All totaled, I disposed of approximately 250 pieces of ordnance at or near the NODA. After two DOE auditors inspected the site, the name of the site was changed from the Hazardous Materials Disposal Area to the Naval Ordnance Disposal Area because they thought the old name was too sensitive, especially during the time when the environmental regulations were just coming on the scene.

Four problems at NODA were first recognized during three operations. The first was the observation that I had driven over a piece of ordnance on the road after I had thoroughly cleared the road. The particular incident occurred during vehicular entry to NODA after a rainstorm. The soil was not particularly wet but I felt the vehicle hit something. I got out and inspected the area and found I had driven over a fuse 3 inches in diameter. An incident similar to this occurred when a Waste Management employee was driving around the firebreak placing danger signs on

the wire fence. His vehicle had hit a 5-inch shell. The next incident occurred during a post-rain inspection of a crater within the NODA. There was a tremendous amount of fragmentation spread around the crater so I started to really look carefully. With the sun to my back I spotted a curved metal surface just off the crater lip, the area where the crater ejecta was located. I carefully uncovered the metal and found three 5-inch artillery shells without ojive fusing. Obviously, a pallet of ordnance had been detonated and some of the ordnance had been kicked out and covered by the ejecta from the blast. The third incident involved the firing of a part of a 2,000-pound bomb. I used a heavy explosive countercharge and determined the safe distance for personnel using standard military tables for cased charges, this distance being approximately 3,500 feet. My assistant and I then moved to that location and fired that shot. The explosion that followed was far in excess of the target ordnance and the countercharge and heavy fragmentation flew over our heads. We returned to ground zero and found four 3-inch shells and one 5-inch shell in place of the bomb component we had fired.

We are continually finding pieces that have worked their way to the surface. Any farmboy who has ever farmed in Idaho knows that rocks grow. I continued explosive disposal up until about four years ago; the environmental regulations have stopped all ordnance disposal at the INEL. It is time that the personnel safety risks associated with ordnance at the INEL are balanced against the spirit of the environmental regulations.

The chemical operations at NODA were many and varied. NODA was separated into two different areas separated by a 50-foot firebreak. One area was used solely for the interim storage of nonradioactive chemicals extracted from the facilities of the INEL. The other area was used for the disposal of materials that were so reactive that they could not be DOT qualified for shipment to a disposal site, if one could be found. Most often, a legal disposal site could not be found. The materials were usually in a severe state of degradation and posed unacceptable health and safety risks to personnel in the plants. The materials were packaged in vermiculite and nonfrangible containers such as cardboard so that if a reaction occurred, fragmentation would not be produced. Often, we would transport the materials as we would an improvised explosive devise. Once we had transported the material to NODA, we would place it into metal garden sheds to await the determination of a place to send it. The extremely hazardous reactives were then placed in a separate area to await disposal in existing bomb craters on the other side of the firebreak. One of our major fears was the ignition of a range fire during disposal. The circular and bisection firebreaks were very effective barriers for this threat. We've had experience where a range fire had been accidentally started near Fire Station II and had burned approximately 800 acres. Hundreds of pieces of ordnance cooked off and exploded while we were trying to extinguish the blaze. That was when we really realized we had a large-area explosive ordnance problem at the INEL. The approximate total quantity of chemicals extracted from INEL facilities was 98,000 pounds, including packaging, most of which was shipped to legal repositories. The facility at Grand View, Idaho, was used often. Only about 7,000 pounds of reactives were processed at NODA, and most of this was finely divided zirconium, hafnium, and zircalloy that was pyrophoric and was therefore burned in buckets and pans. The oxides were then collected and transported to the landfill for final internment.

The primary chemical disposal operations conducted at NODA were remote openings of containers in metal pans for sampling; combustion reactions of reagents that were unsafe to ship, reactive, and possibly explosive; neutralizations; air releases of small quantities of reactants such

as those found in lab bottles; pyrophoric burns that sometimes deflagrated; detonations of those materials that would support a reactive shock wave; and, of course, explosive ordnance disposal.

I developed a number of reactive tests that I would use to determine if the chemicals would support combustion or a reactive shock. I never shot an unknown. My intention was never to explosively disperse any chemical agent to the environment. I had the help of a number of analytical chemists for analysis of unknowns I had remotely sampled. Residues from all chemical reactions, with the exception of the materials detonated, were collected, drummed, or packaged, and sent to the sanitary landfill. Before a chemical was reacted, attempts were made to determine the products of reaction and ensure minimal environmental impact. My self-imposed rule to detonate only CHNO-containing reactives pretty well fulfilled this requirement. There were just a few exceptions because the materials were dangerously explosive. An example was lead azide and lead-contaminated sodium azide. Some materials were reacted in stainless steel reaction chambers, sampled, repackaged, and sent to a repository. The following list of chemicals were disposed of at the NODA:

PEROXIDE FORMERS

Diethyl ether
Diacetylene
Diethylene glycol dimethyl ether
Diisopropyl ether
Potassium metal
Potassium amide
Sodium amide
Dioxane
Furan
Ethylene glycol dimethyl ether
Tetrahydrofuran
Styrene

PYROPHORIC CHEMICALS

Grignard reagent
Nickel Carbonyl
Iron Carbonyl
Alkyl lithium in ether
Sodium
Potassium
Lithium Rubidium
Sodium-Potassium Eutectic Alloy (NAK)
Finely divided metal powders: Al, Mg, Mn, Zr, Hf, Zircalloy
Metal hydrides: Sodium hydride, Potassium hydride, Lithium Aluminum Hydride
Nonmetal hydrides: Diboranes, Phosphine
White and red phosphorus

WATER REACTIVES

Alkali metals
Calcium carbide
Alkali metal hydrides
Grignard reagent
Anhydrous metal halides: Titanium tetra chloride
Barium
Calcium

EXPLOSIVES/EXPLOSIVELY REACTIVE

Nitro compounds
Nitroso compounds
Azides: Lead azide, Sodium azide (note: this is not an explosive)
Copper acetylide
Trinitrotoluene
Composition B (RDX, TNT)
Tritonal (Al, TNT)
Picric Acid (Trinitrophenol)
Metal picrates (Ni, Cu, Fe)
Perchlorates (metal, nonmetal, organic admixtures)
Amatol (Ammonium nitrate/TNT)
Fulminates (Silver and Mercury)
Nitrates (Ammonium nitrate, Calcium nitrate/Oil)
Hydrogen Peroxide (60-90%)
Nitroglycerine
Nitroguanidine
Tetryl
Oxidant/Reductant Admixtures (nitrates, nitric acid/organic, nitric acid/cellulose, iodates, chlorites, chlorates, permanganates)

Magnesium perchlorate/organic admixture
Organic peroxides: dialkyl peroxides, diacyl peroxides, benzoyl peroxide
Dipicryl amine
Dinitrophenylhydrazine
nitrosoguanidine
Perchloric acid/organic admixtures
Metal perchlorates
Artillery shells
Mines
Fuses
Bombs
Disassembled Perchloric Acid Hoods and Blowers and Ducts
Cyclotrimethylenetrinitramine (RDX)
Potassium metal/Potassium superoxide
HMX

Ammoniacal Compounds (nitrogen triiodide)
Chlorite salts of metals
Xylene-Aliquat-Perchloric Acid Admixtures

Appendix B

Fourier Transform Infrared Monitoring of NODA

INTEROFFICE CORRESPONDENCE

Date: June 11, 1992
To: J. B. Sisson, MS 2107
From: G. L. Olson, MS 2213
Subject: FTIR MONITORING OF NODA - GLO-11-92

From June 1-June 5, we conducted open path FTIR measurements at the IRC, RWMC and NODA. The purpose of the testing was to attempt to substantiate or verify an infrared "hot" spot, which was measured at the NODA site in a 1991 flyover by Martin Marietta. (Calibration and operational tests were conducted at IRC and RWMC.) Open path FTIR measurements were deemed appropriate at the NODA site for several reasons: 1) FTIR is sensitive to IR absorbing compounds, 2) measurements can be made along an integrated path (rather than on a point by point basis), which circumvents the problem of hitting or missing "hot" spots, 3) the measurements are real-time, 4) there is no need to collect and handle samples, 5) prior knowledge of the contaminants present is unnecessary. FTIR is sensitive for most organic compounds to the ppm and ppb range. For example, detection limits with the Nicolet system at 0.5 cm⁻¹ resolution for TCE and carbon tetrachloride are less than 10 ppm.

To ensure that the best results possible were obtained, we collected data from the NODA site when the atmospheric conditions were stable. We also tested the instrument to determine if it would detect contaminants if they were present. We accomplished this by obtaining a standard prepared by Larry Lazzarotto, and introducing the known amount of analytes into the beam. The analytes and their concentrations were: 755 ppm carbon tetrachloride, 339 ppm TCE, and 120 ppm chloroform. The FTIR positively identified all of the compounds in the mixture. The FTIR also positively identified emissions from the gas-operated generators during system check-out at the IRC, and from vehicular traffic at the NODA. Ozone, N₂O, methane, ammonia, water and CO₂ were all measured at ambient concentrations.

Aside from positive identification of species from auto exhaust, no contaminants were detected at the NODA. According to others that have used open path FTIR for assessments of Superfund sites, this is not an unexpected result at sites with buried or old waste (personal communication Timothy Minnich, Blasland, Bouck, and Lee, 6-11-92; and David Mickunas, Weston/REAC, 6-11-92). I have included as Attachment 1 a paper by Solinski et al. (1992), that describes FTIR monitoring at an industrial lagoon containing several carcinogens. Although the waste is in a sludge form in an open lagoon (with a layer of water), contaminant emissions are not present above the instrument detection limit.

June 11, 1992
J. B. Sisson
GLO-10-92
Page 2

Attachment 2 shows results of FTIR monitoring at the SDA at the RWMC in 1991 by Robert Kagann of MDA Scientific, Inc. The discussion on SDA results (p. 3) show that no detectable amounts of compounds other than ozone and methanol were measured with the MDA (Kagann, 1991, attachment 2). Methanol was at the detection limit. Importantly, the MDA system is not as sensitive as the Nicolet system, which we used, for two reasons: 1) the MDA uses a 1 cm¹ resolution, compared to Nicolet's 0.5 cm¹ resolution, and 2) the MDA uses only one telescope, which functions as a source and a receiver. This arrangement requires that the beam pass through the beamsplitter twice, resulting in an estimated 75% loss of energy (Bill Herget, personal communication, 5-28-92).

Attachment 3 contains the results of another study at the RWMC, in which Jeff Erickson (EG&G industrial hygienist) monitored employee exposure during drilling operations at the SDA. Five organic species were analyzed: carbon tetrachloride (CTC), methyl chloroform (or 1,1,1-trichloroethane [TCA]), tetrachloroethylene or perchloroethylene (PCE), trichloroethylene (TCE), and methylene chloride or dichloromethane (DCM). The badges used were passive dosimeters, which collected the vapors throughout the exposure time, then were analyzed in the EG&G industrial hygiene laboratory. Results from Erickson's study showed none of the contaminants exceeding 2.5 ppm for an 8-hour time weighted average.

In light of the evidence, the lack of detectable concentrations of organic compounds at NODA or the RWMC is not unusual; however, it would be prudent to spot check these areas using traditional sampling techniques, or using traditional equipment in a way that allows for comparison with the open path technique (e.g., carry the summa canister along the path length measured with the FTIR).

For your information and documentation, I have recorded our daily activities and provided them as Attachment 4. Data from this activity was sent to me by Bill Herget, and is provided as Attachment 5. Thank you again for your support.

mam

Attachments:
As stated

cc: L. J. Johnson, MS 2203
R. P. Breckenridge, MS 2213
Central Files
Letter File

Appendix C

Soil Samples at NODA—Twin City Testing Corporation Laboratory Report

TCT - St. Louis

Consulting Engineers, Scientists and Analytical Services

COPY

1908 Innerbelt Business Center Drive
St. Louis, Missouri 63114-5700
Phone (314) 426-0880
Fax (314) 426-4212

September 24, 1992
9261-00001

Unvalidated Data ☒
Unvalidated Data ☒
FOR EG&G INTERNAL USE ONLY
Date: 9-25-92

Ms. Donna Kirchner
Field Data Coordinator
ERP Administrative Record
and Document Control
EG&G Idaho, Inc.
P.O. Box 1625
Idaho Falls, ID 83415-3904

Re: C92-170021 ERD-TOS-03 Task Order #16

Dear Ms. Kirchner:

Enclosed is a data report for the analysis of volatile organic compounds, PCB's and metals for the soil samples received by TCT-St. Louis under Subcontract No. C92-170021-CN-233-92, (ERD-TOS-03). The samples were collected from the NODA gravel pad at the Idaho National Engineering Laboratory, in an effort to provide characterization data for the determination of possible environmental contamination under the NODA gravel pad.

The EG&G site code, corresponding TCT-St. Louis File I.D., laboratory sample number, and date collected are listed below:

<u>EG&G SITE ID</u>	<u>FILE ID</u>	<u>LAB NUMBER</u>	<u>DATE COLLECTED</u>
NODA92O0305	2O0305	92005508	8-21-92
NODA92M0305	2M0305	92005509	8-21-92
NODA92O0912	2O0912	92005510	8-21-92
NODA92M0912	2M0912	92005511	8-21-92
NODA92O1023	2O1023	92005512	8-21-92
NODA92M1023	2M1023	92005513	8-21-92
NODA92O1627	2O1627	92005514	8-21-92
NODA92M1627	2M1627	92005515	8-21-92
NODA92O2525	2O2525	92005516	8-21-92
NODA92M2525	2M2525	92005517	8-21-92

The samples were received by TCT-ST. Louis on August 28, 1992. A copy of the chain-of-custody was mailed to you on August 31, 1992.

EG&G IDAHO, INC.
C92-170021
ERD-TOS-03; Task Order #16
page 2

CLP-METALS

The samples designated for metals analysis were assigned the SDG number - 2O0305. Quality control (matrix spike and duplicate analyses) was performed on sample 2O0305. All samples were digested and analyzed within the required holding times. A CLP metals package has been assembled and is included.

PCB's

The samples analyzed for PCB's are grouped under the SDG of 2O0305. The samples were analyzed by SW846/8080 and not CLP methodology as requested in the Task Order. The modification to proceed with SW846/8080 was authorized by Rod Grant of EG&G on September 14, 1992. Sample 200305 is designated for quality control and a matrix spike and matrix spike duplicate analysis was performed on this sample. All surrogates are within limits. Sample 2O1627 was diluted 1:10 to allow for accurate quantitation. All samples were extracted and analyzed within holding times. A PCB data package has been assembled and is included.

VOLATILES

The samples for volatile analysis by CLP 2/88 methodology are grouped under the SDG of 2O0305. All surrogate recoveries are within CLP recovery range. A matrix spike and matrix spike duplicate analysis was performed on sample 2O0305. All spike recoveries and RPD's are within CLP limits.

A complete CLP volatiles data package has been assembled and is included.

If you have any questions concerning this data report, please call me at (314) 426-0880.

Sincerely,

Paul J. Smith
Sr. Project Manager

1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EG&G SAMPLE NO.

200305

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005508

Sample wt/vol: 1 (g/ml) G

Lab File ID: >G6004

Level: (low/med) LOW

Date Received: 08/28/92

% Moisture: not dec. 2

Date Analyzed: 09/03/92

Column: (pack/cap) CAP

Dilution Factor: 5

CAS NO.	COMPOUND	CONCENTRATION UNITS:	
		(ug/L or ug/Kg)	ug/Kg
			Q
74-87-3	Chloromethane	51	IU
74-83-9	Bromomethane	51	IU
75-01-4	Vinyl chloride	51	IU
75-00-3	Chloroethane	51	IU
75-09-2	Methylene Chloride	240	I
67-64-1	Acetone	640	I
75-15-0	Carbon Disulfide	25	IU
75-35-4	1,1-Dichloroethene	25	IU
75-34-3	1,1-Dichloroethane	25	IU
540-59-0	1,2-Dichloroethene (total)	25	IU
67-66-3	Chloroform	25	IU
107-06-2	1,2-Dichloroethane	25	IU
78-93-3	2-Butanone	25	IU
71-55-6	1,1,1-Trichloroethane	25	IU
56-23-5	Carbon Tetrachloride	25	IU
108-05-4	Vinyl Acetate	51	IU
75-27-4	Bromodichloromethane	25	IU
78-87-5	1,2-Dichloropropane	25	IU
10061-01-5	cis-1,3-Dichloropropene	25	IU
79-01-6	Trichloroethene	25	IU
124-48-1	Dibromochloromethane	25	IU
79-00-5	1,1,2-Trichloroethane	25	IU
71-43-2	Benzene	25	IU
10061-02-6	trans-1,3-Dichloropropene	25	IU
75-25-2	Bromoform	25	IU
108-10-1	4-Methyl-2-pentanone	51	IU
591-78-6	2-Hexanone	51	IU
127-18-4	Tetrachloroethene	25	IU
79-34-5	1,1,2,2-Tetrachloroethane	25	IU
108-88-3	Toluene	25	IU
108-90-7	Chlorobenzene	25	IU
100-41-4	Ethylbenzene	25	IU
100-42-5	Styrene	25	IU
1330-20-7	Xylene (total)	25	IU

FORM I UDA

1/87 Rev

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EG&G SAMPLE NO.

200305

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005508

Sample wt/vol: 1 (g/ml) g

Lab File ID: >G6004

Level: (low/med) LOW

Date Received: 08/28/92

% Moisture: not dec. 2

Date Analyzed: 09/03/92

Column: (pack/cap) CAP

Dilution Factor: 5

Number TICs found: 0

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.				
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FORM I VOA-TIC

1/87 Rev

PESTICIDE ORGANICS DATA SHEET
1D

EG+6 SAMPLE NO.

200305

Lab Name: TCT-ST. LOUIS

Contract: ERD-T03-03

Lab Code: TCT Case No.: T0503

Sas: SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005508

Sample wt/vol: 15.0(g/ml) g

Lab File ID: NA

Level: (low/med) Low

Date Received: 8-28-92

%Moisture: not dec. 1.5 dec. NA

Date Extracted: 9-4-92

Extraction (SepF/Cont/Sonc) Sonc

Date Analyzed: 9-14-92

GPC Cleanup: (Y/N) N pH 7

Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS:	
12674-11-2	AROCLOP-1016	34 $\mu\text{g/kg}$	L'
11104-26-2	AROCLOP-1221		u
11141-16-5	AROCLOP-1232		u
53463-21-9	AROCLOP-1243		u
12672-29-6	AROCLOP-1248		u
11097-69-1	AROCLOP-1254		u
11096-62-5	AROCLOP-1260	34	u

FORM 1 PEST

1/87 Rev.

ENVIROFORMS/INORGANIC CLP

SAMPLE NO.

1
INORGANIC ANALYSIS DATA SHEET

2M0305

Lab Name: TCT St. Louis

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 2M0305

Matrix (soil/water): SOIL

Lab Sample ID: 92005509

Level (low/med): LOW

Date Received: 08/28/92

% Solids: 98.5

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	7820	—	—	P
7440-36-0	Antimony	1.4	U	N	F
7440-38-2	Arsenic	7.1	—	NS	F
7440-39-3	Barium	162	—	—	P
7440-41-7	Beryllium	1.6	—	—	P
7440-43-9	Cadmium	1.0	U	—	P
7440-70-2	Calcium	5830	—	—	P
7440-47-3	Chromium	26.4	—	—	P
7440-48-4	Cobalt	9.3	B	—	P
7440-50-8	Copper	44.3	—	N*	P
7439-89-6	Iron	16800	—	—	P
7439-92-1	Lead	72.1	—	N	P
7439-95-4	Magnesium	4950	—	—	P
7439-96-5	Manganese	343	—	—	P
7439-97-6	Mercury	0.06	B	—	CV
7440-02-0	Nickel	27.6	—	—	P
7440-09-7	Potassium	3030	—	—	A
7782-49-2	Selenium	0.41	U	NW	F
7440-22-4	Silver	0.28	B	—	F
7440-23-5	Sodium	122	B	—	P
7440-28-0	Thallium	0.67	B	W	F
7440-62-2	Vanadium	19.7	—	—	P
7440-66-6	Zinc	130	—	—	P
	Cyanide		—	—	—

Color Before: BROWN

Clarity Before:

Texture: FINE

Color After: YELLOW

Clarity After:

Artifacts: NO

Comments:

1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EG&G SAMPLE NO.

200912

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005510

Sample wt/vol: 5 (g/ml) G

Lab File ID: >G5989

Level: (low/med) LOW

Date Received: 08/28/92

% Moisture: not dec. 14

Date Analyzed: 09/02/92

Column: (pack/cap) CAP

Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS:		Q
		(ug/L or ug/Kg)	ug/Kg	
74-87-3	Chloromethane	10	IU	
74-83-9	Bromomethane	10	IU	
75-01-4	Vinyl chloride	10	IU	
75-00-3	Chloroethane	10	IU	
75-09-2	Methylene Chloride	150	B	
67-64-1	Acetone	190	B	
75-15-0	Carbon Disulfide	5	IU	
75-35-4	1,1-Dichloroethene	5	IU	
75-34-3	1,1-Dichloroethane	5	IU	
540-59-0	1,2-Dichloroethene (total)	5	IU	
67-66-3	Chloroform	5	IU	
107-06-2	1,2-Dichloroethane	5	IU	
78-93-3	2-Butanone	10	IU	
71-55-6	1,1,1-Trichloroethane	5	IU	
56-23-5	Carbon Tetrachloride	5	IU	
108-05-4	Vinyl Acetate	10	IU	
75-27-4	Bromodichloromethane	5	IU	
78-87-5	1,2-Dichloropropane	5	IU	
10061-01-5	cis-1,3-Dichloropropene	5	IU	
79-01-6	Trichloroethene	5	IU	
124-48-1	Dibromochloromethane	5	IU	
79-00-5	1,1,2-Trichloroethane	5	IU	
71-43-2	Benzene	5	IU	
10061-02-6	trans-1,3-Dichloropropene	5	IU	
75-25-2	Bromoform	5	IU	
108-10-1	4-Methyl-2-pentanone	10	IU	
591-78-6	2-Hexanone	10	IU	
127-18-4	Tetrachloroethene	5	IU	
79-34-5	1,1,2,2-Tetrachloroethane	5	IU	
108-88-3	Toluene	5	IU	
108-90-7	Chlorobenzene	5	IU	
100-41-4	Ethylbenzene	5	IU	
100-42-5	Styrene	5	IU	
1330-20-7	Xylene (total)	5	IU	

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EG&G SAMPLE NO.

200912

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005510

Sample wt/vol: 5 (g/ml) g

Lab File ID: >G5989

Level: (low/med) LOW

Date Received: 08/28/92

% Moisture: not dec. 14

Date Analyzed: 09/02/92

Column: (pack/cap) CAP

Dilution Factor: 1

Number TICs found: 1

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1. 110543	HEXANE	5.40	15	J
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FORM I VOA-TIC

1/87 Rev

PESTICIDE ORGANICS DATA SHEET
1D

EG+G SAMPLE NO.

200912

Lab Name: TCT-ST. LOUIS

Contract: ERD-TOS-03

Lab Code: TCT Case No.: TOS03

Sas: SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005510

Sample wt/vol: 15.0 (g/ml): g

Lab File ID: NA

Level: (low/med): low

Date Received: 8-28-92

%Moisture: not dec. 14.2 dec. NA

Date Extracted: 9-4-92

Extraction (SepF/Cont/Sonc) SONC

Date Analyzed: 9-15-92

GPC Cleanup: (Y/N) N pH 7

Dilution Factor: 1

CONCENTRATION UNITS:

CAS NO.

COMPOUND

12674-11-2	AROCLOF-1016	38	ug/kg	u
11104-26-2	AROCLOF-1221	78		u
11141-16-5	AROCLOF-1232	38		u
53465-21-9	AROCLOF-1242	38		u
12672-29-6	AROCLOF-1246	38		u
11097-69-1	AROCLOF-1254	38		u
11096-62-5	AROCLOF-1260	47		u

FORM 1 PEST

1/87 Rev.

ENVIROFORMS/INORGANIC CLP

SAMPLE NO.

1
INORGANIC ANALYSIS DATA SHEET

2M0912

Lab Name: TCT St. Louis

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 2M0305

Matrix (soil/water): SOIL

Lab Sample ID: 92005511

Level (low/med): LOW

Date Received: 08/28/92

% Solids: 85.8

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	10700	---		P
7440-36-0	Antimony	1.6	U	N	P
7440-38-2	Arsenic	4.5	---	N	P
7440-39-3	Barium	165	---		P
7440-41-7	Beryllium	1.4	---		P
7440-43-9	Cadmium	1.9	---		P
7440-70-2	Calcium	22600	---		P
7440-47-3	Chromium	35.0	---		P
7440-48-4	Cobalt	10.3	B		P
7440-50-8	Copper	21.9	---	N*	P
7439-89-6	Iron	19900	---		P
7439-92-1	Lead	9.5	---	W	P
7439-95-4	Magnesium	8110	---		P
7439-96-5	Manganese	298	---		P
7439-97-6	Mercury	0.27	---		CV
7440-02-0	Nickel	27.3	---		P
7440-09-7	Potassium	2930	---		A
7782-49-2	Selenium	0.47	U	NW	P
7440-22-4	Silver	0.23	U		P
7440-23-5	Sodium	190	B		P
7440-28-0	Thallium	0.47	U	W	P
7440-62-2	Vanadium	35.2	---		P
7440-66-6	Zinc	106	---		P
	Cyanide		---		

Color Before: BROWN

Clarity Before:

Texture: SANDY

Color After: LT.YELLOW

Clarity After:

Artifacts: NO

Comments:

VOLATILE ORGANICS ANALYSIS DATA SHEET

LABORATORY NO.

201023

Lab Name: TCT-ST. LOUIS

Contract: ERDT0503

Lab Code: TCT

Case No.: T0503

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005512

Sample wt/vol: 1 (g/ml) G

Lab File ID: >G6002

Level: (low/med) LOW

Date Received: 08/28/92

% Moisture: not dec. 0

Date Analyzed: 09/03/92

Column: (pack/cap) CAP

Dilution Factor: 5

CAS NO.	COMPOUND	CONCENTRATION UNITS:	
		(ug/L or ug/Kg)	ug/Kg
74-87-3	Chloromethane	50	U
74-83-9	Bromomethane	50	U
75-01-4	Vinyl chloride	50	U
75-00-3	Chloroethane	50	U
75-09-2	Methylene Chloride	260	
67-64-1	Acetone	500	
75-15-0	Carbon Disulfide	25	U
75-35-4	1,1-Dichloroethene	25	U
75-34-3	1,1-Dichloroethane	25	U
540-59-0	1,2-Dichloroethene (total)	25	U
67-66-3	Chloroform	25	U
107-06-2	1,2-Dichloroethane	25	U
78-93-3	2-Butanone	11	J
71-55-6	1,1,1-Trichloroethane	25	U
56-23-5	Carbon Tetrachloride	25	U
108-05-4	Vinyl Acetate	50	U
75-27-4	Bromodichloromethane	25	U
78-87-5	1,2-Dichloropropane	25	U
10061-01-5	cis-1,3-Dichloropropene	25	U
79-01-6	Trichloroethene	5	J
124-48-1	Dibromochloromethane	25	U
79-00-5	1,1,2-Trichloroethane	25	U
71-43-2	Benzene	8	J
10061-02-6	trans-1,3-Dichloropropene	25	U
75-25-2	Bromoform	25	U
108-10-1	4-Methyl-2-pentanone	50	U
591-78-6	2-Hexanone	50	U
127-18-4	Tetrachloroethene	25	U
79-34-5	1,1,2,2-Tetrachloroethane	25	U
108-88-3	Toluene	6	J
108-90-7	Chlorobenzene	25	U
100-41-4	Ethylbenzene	25	U
100-42-5	Styrene	25	U
1330-20-7	Xylene (total)	25	U

FORM I VOA

1/87 Rev

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EG&G SAMPLE NO.

201023

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005512

Sample wt/vol: 1 (g/ml) g

Lab File ID: >G6002

Level: (low/med) LOW

Date Received: 08/28/92

% Moisture: not dec. 0

Date Analyzed: 09/03/92

Column: (pack/cap) CAP

Dilution Factor: 5

Number TICs found: 0

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.				
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FORM 1 VOA-TIC

1/87 Rev

PESTICIDE ORGANICS DATA SHEET
1D

EG-6 SAMPLE NO.

201023

Lab Name: TCT-ST. LOUIS

Contract: ERD-T03-03

Lab Code: TCT Case No.: T0503

Sas: SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005512

Sample wt/vol: 15.0(g/ml) g

Lab File ID: NA

Level: (low/med) low

Date Received: 8-28-92

%Moisture: not dec. 0.2 dec. NA

Date Extracted: 9-4-92

Extraction (SepF/Cont/Sonc): SONC

Date Analyzed: 9-15-92

GPC Cleanup: (Y/N) N pH 7

Dilution Factor: 1

CAS NO.

COMPOUND

CONCENTRATION UNITS:

CAS NO.	COMPOUND	CONCENTRATION UNITS:
12674-11-2	AROCLOF-1016	33 ug/kg 11
11104-28-2	AROCLOF-1221	67 11
11141-16-5	AROCLOF-1232	33 11
11461-21-9	AROCLOF-1241	33 11
12672-29-6	AROCLOF-1248	33 11
11097-69-1	AROCLOF-1254	33 11
11096-82-5	AROCLOF-1260	318 1

FORM 1 PEST

1/87 Rev.

ENVIROFORMS/INORGANIC CLP

SAMPLE NO.

1
INORGANIC ANALYSIS DATA SHEET

2M1C23

Lab Name: TCT St. Louis

Contract: ERDTOS02

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 2M0305

Matrix (soil/water): SOIL

Lab Sample ID: 92005513

Level (low/med): LOW

Date Received: 08/28/92

% Solids: 99.8

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	6230			P
7440-36-0	Antimony	1.4	U	N	F
7440-38-2	Arsenic	4.2		NS	F
7440-39-3	Barium	98.8			P
7440-41-7	Beryllium	1.0			P
7440-43-9	Cadmium	1.0			P
7440-70-2	Calcium	22000			P
7440-47-3	Chromium	16.6			P
7440-48-4	Cobalt	7.0	B		P
7440-50-8	Copper	37.1		N*	P
7439-89-6	Iron	12700			P
7439-92-1	Lead	6.9		W	F
7439-95-4	Magnesium	5250			P
7439-96-5	Manganese	222			P
7439-97-6	Mercury	0.06	B		CV
7440-02-0	Nickel	20.4			P
7440-09-7	Potassium	2000			A
7782-49-2	Selenium	0.40	U	NW	F
7440-22-4	Silver	0.20	U		F
7440-23-5	Sodium	135	B		P
7440-28-0	Thallium	0.40	B	W	F
7440-62-2	Vanadium	21.2			P
7440-66-6	Zinc	120			P
	Cyanide				

Color Before: BROWN

Clarity Before:

Texture: SANDY

Color After: LT.YELLOW

Clarity After:

Artifacts: NO

Comments:

1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EG&G SAMPLE NO.

201627

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005514

Sample wt/Vol: 2.5 (g/ml) G

Lab File ID: >G6003

Level: (low/med) LOW

Date Received: 08/28/92

% Moisture: not dec. 0

Date Analyzed: 09/03/92

Column: (pack/cap) CAP

Dilution Factor: 2

CAS NO.	COMPOUND	CONCENTRATION UNITS:		Q
		(ug/L or ug/Kg)	ug/Kg	
74-87-3	Chloromethane	20	1U	
74-83-9	Bromomethane	20	1U	
75-01-4	Vinyl chloride	20	1U	
75-00-3	Chloroethane	20	1U	
75-09-2	Methylene Chloride	280	1	
67-64-1	Acetone	260	1	
75-15-0	Carbon Disulfide	10	1U	
75-35-4	1,1-Dichloroethene	10	1U	
75-34-3	1,1-Dichloroethane	10	1U	
540-59-0	1,2-Dichloroethene (total)	10	1U	
67-66-3	Chloroform	10	1U	
107-06-2	1,2-Dichloroethane	10	1U	
78-93-3	2-Butanone	10	1U	
71-55-6	1,1,1-Trichloroethane	10	1U	
56-23-5	Carbon Tetrachloride	10	1U	
108-05-4	Vinyl Acetate	20	1U	
75-27-4	Bromodichloromethane	10	1U	
78-87-5	1,2-Dichloropropane	10	1U	
10061-01-5	cis-1,3-Dichloropropene	10	1U	
79-01-6	Trichloroethene	10	1U	
124-48-1	Dibromochloromethane	10	1U	
79-00-5	1,1,2-Trichloroethane	10	1U	
71-43-2	Benzene	10	1U	
10061-02-6	trans-1,3-Dichloropropene	10	1U	
75-25-2	Bromoform	10	1U	
108-10-1	4-Methyl-2-pentanone	20	1U	
591-78-6	2-Hexanone	20	1U	
127-18-4	Tetrachloroethene	10	1U	
79-34-5	1,1,2,2-Tetrachloroethane	10	1U	
108-88-3	Toluene	10	1U	
108-90-7	Chlorobenzene	10	1U	
100-41-4	Ethylbenzene	10	1U	
100-42-5	Styrene	10	1U	
1330-20-7	Xylene (total)	10	1U	

FORM I UOA

1/87 Rev

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

E & G SAMPLE NO.

201627

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005514

Sample wt/vol: 2.5 (g/ml) g

Lab File ID: >G6003

Level: (low/med) LOW

Date Received: 08/28/92

% Moisture: not dec. 0

Date Analyzed: 09/03/92

Column: (pack/cap) CAP

Dilution Factor: 2

Number TICs found: 2

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1. 110543	HEXANE	5.49	24	J
2. 124185	DECANE	19.85	12	J
3.				
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FORM I UDA-TIC

1/87 Rev

PESTICIDE ORGANICS DATA SHEET
1D

EG+6 SAMPLE NO.

201627

2

Lab Name: TCT-ST. LOUIS

Contract: ERD-TOS-03

Lab Code: TCT Case No.: TOS03

Sas:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005514

Sample wt/vol: 15.0(g/ml) g

Lab File ID: NA

Level: (low/med) Low

Date Received: 8-28-92

Moisture: not dec. 0.3 dec. NA

Date Extracted: 9-4-92

Extraction (SepF/Cont/Sonc) Sonc

Date Analyzed: 9-15-92

GPC Cleanup: (Y/N) N pH 7

Dilution Factor: 10

CONCENTRATION UNITS:

CAS NO.

COMPOUND

12674-11-2	AROCLOF-1016	330	ug/kg	U
11104-28-2	AROCLOF-1221	670		U
11141-16-5	AROCLOF-1232	330		U
53463-21-9	AROCLOF-1241	530		U
12672-29-6	AROCLOF-1248	330		U
11097-69-1	AROCLOF-1254	330		U
11096-82-5	AROCLOF-1260	2251		

FORM 1 PEST

1/87 Rev.

ENVIROFORMS/INORGANIC CLP

SAMPLE NO.

1
INORGANIC ANALYSIS DATA SHEET

2M1627

Lab Name: TCT St. Louis

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 2M0305

Matrix (soil/water): SOIL

Lab Sample ID: 92005515

Level (low/med): LOW

Date Received: 08/28/92

% Solids: 99.7

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	C	M
7429-90-5	Aluminum	6300	—	—	P
7440-36-0	Antimony	1.4	U	N	F
7440-38-2	Arsenic	7.9	—	NS	F
7440-39-3	Barium	95.9	—	—	P
7440-41-7	Beryllium	1.0	—	—	P
7440-43-9	Cadmium	1.0	U	—	P
7440-70-2	Calcium	20100	—	—	P
7440-47-3	Chromium	18.7	—	—	P
7440-48-4	Cobalt	6.4	B	—	P
7440-50-8	Copper	15.2	—	N*	P
7439-89-6	Iron	13300	—	—	P
7439-92-1	Lead	12.2	—	W	F
7439-95-4	Magnesium	5020	—	—	P
7439-96-5	Manganese	215	—	—	P
7439-97-6	Mercury	0.36	—	—	CV
7440-02-0	Nickel	18.5	—	—	P
7440-09-7	Potassium	1630	—	—	A
7782-49-2	Selenium	4.0	U	N	F
7440-22-4	Silver	0.20	U	—	F
7440-23-5	Sodium	145	B	—	P
7440-28-0	Thallium	0.40	U	W	F
7440-52-2	Vanadium	26.3	—	—	P
7440-66-6	Zinc	71.6	—	—	P
	Cyanide		—	—	—

Color Before: BROWN

Clarity Before:

Texture: SANDY

Color After: LT. YELLOW

Clarity After:

Artifacts: NO

Comments:

VOLATILE ORGANICS ANALYSIS DATA SHEET

202525

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005516

Sample wt/vol: 1 (g/ml) G

Lab File ID: >G5998

Level: (low/med) LOW

Date Received: 08/28/92

% Moisture: not dec. 15

Date Analyzed: 09/03/92

Column: (pack/cap) CAP

Dilution Factor: 5

		CONCENTRATION UNITS:		
		(ug/L or ug/Kg) ug/Kg		Q
CAS NO.	COMPOUND			
74-87-3	Chloromethane	59	U	
74-83-9	Bromomethane	59	U	
75-01-4	Vinyl chloride	59	U	
75-00-3	Chloroethane	59	U	
75-09-2	Methylene Chloride	500		
67-64-1	Acetone	1200		
75-15-0	Carbon Disulfide	29	U	
75-35-4	1,1-Dichloroethene	29	U	
75-34-3	1,1-Dichloroethane	29	U	
540-59-0	1,2-Dichloroethene (total)	29	U	
67-66-3	Chloroform	29	U	
107-06-2	1,2-Dichloroethane	29	U	
78-93-3	2-Butanone	59	U	
71-55-6	1,1,1-Trichloroethane	29	U	
56-23-5	Carbon Tetrachloride	29	U	
108-05-4	Vinyl Acetate	59	U	
75-27-4	Bromodichloromethane	29	U	
78-87-5	1,2-Dichloropropane	29	U	
10061-01-5	cis-1,3-Dichloropropene	29	U	
79-01-6	Trichloroethene	29	U	
124-48-1	Dibromochloromethane	29	U	
79-00-5	1,1,2-Trichloroethane	29	U	
71-43-2	Benzene	29	U	
10061-02-6	trans-1,3-Dichloropropene	29	U	
75-25-2	Bromoform	29	U	
108-10-1	4-Methyl-2-pentanone	59	U	
591-78-6	2-Hexanone	59	U	
127-18-4	Tetrachloroethene	29	U	
79-34-5	1,1,2,2-Tetrachloroethane	29	U	
108-88-3	Toluene	29	U	
108-90-7	Chlorobenzene	29	U	
100-41-4	Ethylbenzene	29	U	
100-42-5	Styrene	29	U	
1330-20-7	Xylene (total)	29	U	

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EG&G SAMPLE NO.

202525

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 92005516

Sample wt/vol: 1 (g/ml) g

Lab File ID: >G5998

Level: (low/med) LOW

Date Received: 08/28/92

% Moisture: not dec. 15

Date Analyzed: 09/03/92

Column: (pack/cap) CAP

Dilution Factor: 5

Number TICs found: 1

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1. 110543	HEXANE	5.51	60	J
2.				
3.				
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FORM I UOA-TIC

1/87 Rev

PESTICIDE ORGANICS DATA SHEET
1D

EG+G SAMPLE NO.

202525

Lab Name: TCT-ST. LOUIS

Contract: ERD-TOS-03

Lab Code: TCT Case No.: TOS03

Sas: SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: 9205516

Sample wt/vol: 15.0 (g/ml) g

Lab File ID: NA

Level: (low/med) LOW

Date Received: 8-28-92

%Moisture: not dec. 15.0 dec. NA

Date Extracted: 9-4-92

Extraction (SepF/Cont/Sonc) SONC

Date Analyzed: 9-15-92

GPC Cleanup: (Y/N) N pH 7

Dilution Factor: 1

CONCENTRATION UNITS:

CAS NO.	COMPOUND	
12674-11-2	AROCLOL-1016	39 ug/kg U
11104-28-2	AROCLOF-1221	79 U
11141-16-5	AROCLOL-1232	39 U
53469-21-9	AROCLOL-1242	39 U
12672-29-6	AROCLOL-1248	39 U
11097-69-1	AROCLOL-1254	39 U
11096-82-5	AROCLOL-1260	78 U

FORM 1 PEST

1/87 Rev.

ENVIROFORMS/INORGANIC CLP

SAMPLE NO.

1
INORGANIC ANALYSIS DATA SHEET

2M2525

Lab Name: TCT St. Louis

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 2MO305

Matrix (soil/water): SOIL

Lab Sample ID: 92005517

Level (low/med): LOW

Date Received: 08/28/92

% Solids: 85.0

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	11900	---	---	P
7440-36-0	Antimony	2.1	B	N	F
7440-38-2	Arsenic	9.1	---	N+	F
7440-39-3	Barium	224	---	---	P
7440-41-7	Beryllium	2.1	---	---	P
7440-43-9	Cadmium	2.1	---	---	P
7440-70-2	Calcium	14900	---	---	P
7440-47-3	Chromium	30.6	---	---	P
7440-48-4	Cobalt	11.3	B	---	P
7440-50-8	Copper	33.6	---	N*	P
7439-89-6	Iron	20900	---	---	P
7439-92-1	Lead	16.9	---	W	F
7439-95-4	Magnesium	9530	---	---	P
7439-96-5	Manganese	386	---	---	P
7439-97-6	Mercury	0.09	B	---	CV
7440-02-0	Nickel	35.3	---	---	P
7440-09-7	Potassium	2950	---	---	A
7782-49-2	Selenium	4.7	U	N	F
7440-22-4	Silver	0.24	B	---	F
7440-23-5	Sodium	192	B	---	P
7440-28-0	Thallium	0.49	B	W	F
7440-62-2	Vanadium	30.8	---	---	P
7440-66-6	Zinc	158	---	---	P
	Cyanide		---	---	---

Color Before: BROWN

Clarity Before:

Texture: FINE

Color After: LT.YELLOW

Clarity After:

Artifacts: NO

Comments:

2B
SOIL VOLATILE SURROGATE RECOVERY

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Level:(low/med) LOW

	EG&G	S1	S2	S3	S4	TOT
	SAMPLE NO.	(TOL)#	(BFB)#	(DCE)#	()#	OUT
01	VBLK01	108	92	93		0
02	200912	105	94	98		0
03						
04						
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06						
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QC LIMITS

S1 (TOL) = Toluene-d8 (81-117)
 S2 (BFB) = 4-Bromofluorobenzene (74-121)
 S3 (DCE) = 1,2-Dichloroethane-d4 (70-121)

Column to be used to flag recovery values
 * Values outside of contract required QC limits
 D Surrogates diluted out

2B
SOIL VOLATILE SURROGATE RECOVERY

Lab Name: TCT-ST. LOUIS

Contract: ERDTOSC3

Lab code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Level:(low/med) LOW

	EG&G	S1	S2	S3	S4	TOT
	SAMPLE NO.	(TOL)#	(BFB)#	(DCE)#	()#	OUT
01	VBLK02	95	99	98		0
02	202525	100	99	104		0
03	200305MS	107	90	103		0
04	200305MSD	103	96	106		0
05	201023	100	96	111		0
06	201627	111	89	112		0
07	200305	100	95	113		0
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QC LIMITS

S1 (TOL) = Toluene-d8 (81-117)
 S2 (BFB) = 4-Bromofluorobenzene (74-121)
 S3 (DCE) = 1,2-Dichloroethane-d4 (70-121)

Column to be used to flag recovery values
 * Values outside of contract required QC limits
 D Surrogates diluted out

SOIL PESTICIDE SURROGATE RECOVERY

Lab Name: TGT- ST. LOUIS

Contract: ERD-705-03

Lab Code: TCT

Case No.: T0503

SMS No.: _____

SDC No.: 200305

Level: (low/med) LOW

	EGRE SAMPLE NO.	S1 (DBC) I	OTHER
01	P&A 763	95	
02	LCS 7123	96	
03	200305	97	
04	I (ms)	98	
05	I (ms0)	95	
06	200912	95	
07	201023	97	
08	201627 (1:10)	107	
09	202325	94	
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ADVISORY
QC LIMITS
(20-150)

S1 (DBC) = Dibutylchlorodate

I Column to be used to flag recovery values

• Values outside of QC limits

D Surrogates diluted out

3B
SOIL VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix Spike - EG&G Sample No.: 92005508

Level: (low/med) LOW

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
1,1-Dichloroethene	50	0	51	102	59-172
Trichloroethene	50	0	46	91	62-137
Chlorobenzene	50	0	47	93	60-133
Toluene	50	0	47	94	59-139
Benzene	50	0	46	91	66-142

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	REC.
1,1-Dichloroethene	50	50	100	1	22	59-172
Trichloroethene	50	46	92	1	24	62-137
Chlorobenzene	50	50	101	8	21	60-133
Toluene	50	49	99	5	21	59-139
Benzene	50	48	96	6	21	66-142

column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD: 0 out of 5 outside limits

Spike Recovery: 0 out of 10 outside limits

COMMENTS: _____

Lab Name: TCT- ST. LOUIS Contract: ELD-TLS-Q3
 Matrix Spike - EG-G Sample No.: 200305 CASE No.: T0303 SDG No.: 200305

COMPOUND	SPIKE ADDED	SAMPLE CONC.	MS CONC.	MS REC	QC LIMITS REC.
AR-1248	337 ug/kg	0	337 ug/kg	107	
			386	114	
			RM 9-17-92	RM 9-17-92	

COMPOUND	SPIKE ADDED	MSD CONC.	MSD REC	RPD	QC LIMITS RPD	REC.
AR-1248	337 ug/kg	337 ug/kg	337	107		
		334	334	114		
		RM 9-17-92	RM 9-17-92	RM 9-17-92		
				9-17-92		

† Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD: _____ out of _____ outside limits
 Spike Recovery: _____ out of _____ outside limits

COMMENTS: _____

VOLATILE METHOD BLANK SUMMARY

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Lab File ID: >G5984

Lab Sample ID: MSA2270

Date Analyzed: 09/02/92

Time Analyzed: 09:39

Matrix: (soil/water) SOIL

Level: (low/med) LOW

Instrument ID: 59704G

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EG&G SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
1	200912	92005510	>G5989	14:50
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COMMENTS:

page ___ of ___.

FORM IV VOA

1/87 Rev.

1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EG&G SAMPLE NO.

UBLK01

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: MSA 2270

Sample wt/vol: 5 (g/ml) G

Lab File ID: >G5984

Level: (low/med) LOW

Date Received:

% Moisture: not dec.

Date Analyzed: 09/02/92

Column: (pack/cap) CAP

Dilution Factor: 1

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NO.	COMPOUND		Q
74-87-3	Chloromethane	10	IU
74-83-9	Bromomethane	10	IU
75-01-4	Vinyl chloride	10	IU
75-00-3	Chloroethane	5	B
75-09-2	Methylene Chloride	6	JB
67-64-1	Acetone	5	IU
75-15-0	Carbon Disulfide	5	IU
75-35-4	1,1-Dichloroethane	5	IU
75-34-3	1,1-Dichloroethane	5	IU
540-59-0	1,2-Dichloroethane (total)	5	IU
67-66-3	Chloroform	5	IU
107-06-2	1,2-Dichloroethane	10	IU
78-93-3	2-Butanone	2	JB
71-55-6	1,1,1-Trichloroethane	5	IU
56-23-5	Carbon Tetrachloride	10	IU
108-05-4	Vinyl Acetate	5	IU
75-27-4	Bromodichloromethane	5	IU
78-87-5	1,2-Dichloropropane	5	IU
10061-01-5	cis-1,3-Dichloropropene	5	IU
79-01-6	Trichloroethene	5	IU
124-48-1	Dibromochloromethane	5	IU
79-00-5	1,1,2-Trichloroethane	5	IU
71-43-2	Benzene	5	IU
10061-02-6	trans-1,3-Dichloropropene	5	IU
75-25-2	Bromoform	10	IU
108-10-1	4-Methyl-2-pentanone	10	IU
591-78-6	2-Hexanone	5	IU
127-18-4	Tetrachloroethene	5	IU
79-34-5	1,1,2,2-Tetrachloroethane	5	IU
108-88-3	Toluene	5	IU
108-90-7	Chlorobenzene	5	IU
100-41-4	Ethylbenzene	5	IU
100-42-5	Styrene	5	IU
1330-20-7	Xylene (total)		

FORM I UOA

1/87 Rev

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EG&G SAMPLE NO.

UBLK01

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: MSA 2270

Sample wt/vol: 5 (g/ml) g

Lab File ID: >G5984

Level: (low/med) LOW

Date Received:

% Moisture: not dec.

Date Analyzed: 09/02/92

Column: (pack/cap) CAP

Dilution Factor: 1

Number TICs found: 0

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.				
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FORM I UOA-TIC

1/87 Rev

4A
VOLATILE METHOD BLANK SUMMARY

Lab Name: TCT-ST. LOUIS Contract: ERDTOS03
 Lab code: TCT Case No.: TOS03 SAS No.: SDG No.: 200305
 Lab File ID: >G5997 Lab Sample ID: MSA2272
 Date Analyzed: 09/03/92 Time Analyzed: 11:32
 Matrix: (soil/water) SOIL Level:(low/med) LOW
 Instrument ID: 59704G

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EG&G SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
1	202525	92005516	>G5998	12:20
2	200305MS	92005508	>G6000	13:40
3	200305MSD	92005508	>G6001	14:20
4	201023	92005512	>G6002	14:59
5	201627	92005514	>G6003	15:39
6	200305	92005508	>G6004	16:26
8				
9				
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COMMENTS: _____

page ___ of ___

FORM IV VOA

1/87 Rev.

1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

VLK02

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: MSA2272

Sample wt/vol: 5 (g/ml) G

Lab File ID: >G5997

Level: (low/med) LOW

Date Received:

% Moisture: not dec.

Date Analyzed: 09/03/92

Column: (pack/cap) CAP

Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS:		Q
		(ug/L or ug/Kg)	ug/Kg	
74-87-3	Chloromethane	10	U	
74-83-9	Bromomethane	10	U	
75-01-4	Vinyl chloride	10	U	
75-00-3	Chloroethane	10	U	
75-09-2	Methylene Chloride	5	U	
67-64-1	Acetone	10	U	
75-15-0	Carbon Disulfide	5	U	
75-35-4	1,1-Dichloroethene	5	U	
75-34-3	1,1-Dichloroethane	5	U	
540-59-0	1,2-Dichloroethene (total)	5	U	
67-66-3	Chloroform	5	U	
107-06-2	1,2-Dichloroethane	5	U	
78-93-3	2-Butanone	10	U	
71-55-6	1,1,1-Trichloroethane	5	U	
56-23-5	Carbon Tetrachloride	5	U	
108-05-4	Vinyl Acetate	10	U	
75-27-4	Bromodichloromethane	5	U	
78-87-5	1,2-Dichloropropane	5	U	
10061-01-5	cis-1,3-Dichloropropene	5	U	
79-01-6	Trichloroethene	5	U	
124-48-1	Dibromochloromethane	5	U	
79-00-5	1,1,2-Trichloroethane	5	U	
71-43-2	Benzene	5	U	
10061-02-6	trans-1,3-Dichloropropene	5	U	
75-25-2	Bromoform	5	U	
108-10-1	4-Methyl-2-pentanone	10	U	
591-78-6	2-Hexanone	10	U	
127-18-4	Tetrachloroethene	5	U	
79-34-5	1,1,2,2-Tetrachloroethane	5	U	
108-88-3	Toluene	5	U	
108-90-7	Chlorobenzene	5	U	
100-41-4	Ethylbenzene	5	U	
100-42-5	Styrene	5	U	
1330-20-7	Xylene (total)	5	U	

FORM I VOA

1/87 Rev

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EG&G SAMPLE NO.

UBLK02

Lab Name: TCT-ST. LOUIS

Contract: ERDTOS03

Lab Code: TCT

Case No.: TOS03

SAS No.:

SDG No.: 200305

Matrix: (soil/water) SOIL

Lab Sample ID: MSA2272

Sample wt/vol: 5 (g/ml) g

Lab File ID: >G5997

Level: (low/med) LOW

Date Received:

% Moisture: not dec.

Date Analyzed: 09/03/92

Column: (pack/cap) CAP

Dilution Factor: 1

Number TICs found: 1

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1. 593759	ISOCYANOMETHANE	3.68	7	JB
2.				
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FORM 1 UOA-TIC

1/87 Rev

PEST CODE METHOD BLANK SUMMARY

: TCT - ST. LOUIS Contract: ERD-TOS-03
 : TCT Case No.: TOS03 SAS No.: _____ SDC No.: 200305
 Lab ID: MB-A 7123 Lab File ID: _____
 Soil/water: SOIL Level: (low/hed) NA
 Extracted: 9-4-92 Extraction: (Sap/Cont/Soil) Soil
 Analyzed (1): 9-14-92 Date Analyzed (2): _____
 Analyzed (1): 21:59 Time Analyzed (2): _____
 Instrument ID (2): _____
 GC Column ID (1): SP2250/2401 GC Column ID (2): _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EG+G SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED 1	DATE ANALYZED 2
01	LC5 7123	LC5 7123	9-14-92	
02	200305	92006508		
03	(ms)	(ms)		
04	(msd)	(msd)	9-15-92	
05	200912	5510		
06	201023	5512		
07	201627 (1:10)	5514 (1:10)		
08	202525	5516		
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Appendix D

**Soil Gas Samples at NODA—
Northeast Research Institute Report**

NORTHEAST RESEARCH INSTITUTE, INC.

November 23, 1992

Mr. Buck Sisson
EG&G
MS-2107
1955 Fremont Avenue
Idaho Falls, Idaho 83415

Re: NERI Project No. 1724

Dear Mr. Sisson:

Northeast Research Institute, Inc. (NERI) has completed a PETREX soil gas investigation for EG&G at the NODA Site located in Idaho Falls, Idaho. The objectives of the PETREX investigation was to provide a test survey to demonstrate the utility of the PETREX Technology in locating and identifying volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs) present in an infrared anomaly identified on the NODA Site. Inasmuch as only five (5) PETREX Collectors were employed in this test survey, instead of a typical grid array of collectors, no attempt was made to map the location of a potential plume of contaminants present on the site in low quantities.

Five PETREX Collectors, each containing two wires, were deployed on the NODA Site. Several VOCs were detected, at very low levels (low mass spectrometer ion counts) using the patented and proprietary PETREX Technology. These VOCs were first identified using pyrolysis/mass spectrometry (Py/MS - First Wire Analysis) and subsequently pyrolysis/gas chromatography/mass spectrometry (Py/GC/MS - Second Wire Analysis). It should be noted that although only two (2) Py/GC/MS analyses were budgeted in this demonstration project, Py/GC/MS analysis was conducted on all of the PETREX Collectors returned to NERI after placement on the test site. The Py/GC/MS analysis technique is applied when complex matrices are encountered, thus, creating a summing of similar fragment ions or when the suspected contaminants are found to be present in very low concentrations. The gas chromatographic separation permits elution of isomers or homologous chemical species which provides the mass spectrometer to detect lower concentrations of pollutants than would be possible in a matrix containing a number of individual compounds.

RESULTS OF PETREX PYROLYSIS/MASS SPECTROMETRIC ANALYSIS

The PETREX Fingerprint Technology (Py/MS Analysis of PETREX Sensors) is a rapid, very sensitive, accurate, and first screening technique that has been utilized in more than 1,000 environmental surveys in twenty-seven countries.

309 Farmington Avenue, Suite A-100 Farmington, Connecticut 06032-1943
Telephone (203) 677-9666 FAX (203) 677-7008

The following list of mass spectral fragments were used by NERI's scientific personnel in reading PETREX Fingerprint mass spectra obtained during the Py/MS analysis of collectors placed on the NODA Site:

Benzene: - 78, 51, 52, 50, 77

Toluene: - 91, 02, 65, 39, 63

Tetrachloroethylene (PCE): - 166, 164, 129, 131, 168

F11 - Trichlorofluoromethane: - 101, 103, 66, 105, 47

Using the key mass spectral fragments listed above, the Py/MS Fingerprint Spectra indicated that the following VOCs are most likely present in the PETREX Collectors which we analyzed following their removal from the NODA site and return to NERI's laboratories:

Travel Blank - Wire # 5

PETREX Collector # 5 was designated as a travel blank. The collector designated as a travel blank is used for quality control purposes. This collector is from the same production lot as the other collectors used in the survey. The travel blank is treated as a normal collector with one exception. This collector is never opened; is placed in the ground with its cap sealed, retrieved, shipped to NERI's laboratories for analysis, and analyzed together with the other collectors. The travel blank, thus, provides information pertaining to background contamination, and will, if not properly handled become contaminated. If the travel blanks used in the field are found to be contaminated an intensive investigation is undertaken to determine the cause of such contamination.

Figure 1 is the reconstructed mass spectrum obtained during the Py/MS analysis of the Travel Blank (Wire # 5). Table I contains specific data obtained during the Py/MS analysis of the Travel Blank and the 4 PETREX Collectors placed on the site.

Figure 1. Py/MS spectrum obtained from the Travel Blank (PETREX Collector #5 - First Wire Analysis)

TABLE I

COMPARISON OF PYROLYSIS/MASS SPECTRAL DATA OBTAINED DURING THE ANALYSIS OF PETREX COLLECTORS PLACED ON THE NODA SITE

WIRE NUMBER	1	2	3	4	5
NUMBER OF PEAKS	111	108	93	86	40
AMU MAXIMUM PEAK (> 45)	82	82	56	56	46
ION COUNT MAXIMUM PEAK (> 45)	58,504	12,800	5,821	14,176	20,593
TOTAL ION COUNT	1,116,994	809,114	709,699	670,019	701,562
TOTAL IONS > MASS 45	251,099	148,018	70,796	98,984	31,691

A study of the data contained in Figure 1 and Table I indicated a total of 40 mass spectral fragments are present in the Travel Blank having a total ion count of 701,562 of which an ion count of only 31,691 is associated with mass fragments greater than mass 45. The maximum peak greater than mass 45 is the fragment at mass 46 which has an ion count of 20,593. It has, therefore, been shown that the Travel Blank is essentially free of fragments which are indicators for common environmental pollutants found in soil and groundwater.

Pyrolysis/Mass Spectrometry (PETREX Collector # 1 - First Wire Analysis)

Figure 2 is the mass spectrum obtained during the Py/MS analysis of PETREX Collector # 1 First Wire Analysis.

Figure 2. Py/MS spectrum obtained from PETREX Collector # 1 First Wire Analysis indicating the presence of benzene, toluene, and tetrachloroethylene

The information presented in Table I indicated a total of 111 mass spectral fragments are present in PETREX Collector # 1 - First Wire Analysis having a total ion count of 1,116,994 of which an ion count of only 251,099 is associated with mass fragments greater than mass 45. The maximum peak greater than mass 45 is the fragment at mass 82 which has an ion count of 58,504. The second largest fragment greater than mass 45 is at mass 207 which is due to column bleed. The Py/MS spectrum obtained during this First Wire Analysis shows the presence of very low concentrations of benzene, toluene (benzene, methyl-), and tetrachloroethylene (PCE).

Pyrolysis/Mass Spectrometry (PETREX Collector # 2 - First Wire Analysis)

Figure 3 is the mass spectrum obtained during the Py/MS analysis of PETREX Collector # 2 - First Wire Analysis. The information presented in Table I indicated a total of 108 mass spectral fragments are present in PETREX Collector # 2 - First Wire Analysis having a total ion count of 809,114 of which an ion count of only 148,018 is associated with mass fragments greater than mass 45. The maximum peak greater than mass 45 is the fragment at mass 82 which has an ion count of 12,800. The second largest fragment greater than mass 45 is at mass 207 which is due to column bleed. The Py/MS spectrum obtained during this First Wire Analysis shows the presence of very low concentrations of benzene, toluene (benzene, methyl-), and tetrachloroethylene (PCE).

Figure 3. Py/MS spectrum obtained from PETREX Collector # 2 First Wire Analysis indicating the presence of benzene, toluene, and tetrachloroethylene

Pyrolysis/Mass Spectrometry (PETREX Collector # 3 - First Wire Analysis)

Figure 4 is the mass spectrum obtained during the Py/MS analysis of PETREX Collector # 3 - First Wire Analysis. The information presented in Table I indicated a total of 93 mass spectral fragments are present in PETREX Collector # 3 - First Wire Analysis having a total ion count of 709,699 of which an ion count of only 70,796 is associated with mass fragments greater than mass 45. The maximum peak greater than mass 45 is the fragment at mass 56 which has an ion count of 5,821. The second largest fragment greater than mass 45 is at mass 207 which is due to column bleed. The Py/MS spectrum obtained during this First Wire Analysis shows the presence of very low concentrations of benzene, toluene (benzene, methyl-), and tetrachloroethylene (PCE).

Figure 4. Py/MS spectrum obtained from PETREX Collector # 3 First Wire Analysis indicating the presence of benzene, toluene, and tetrachloroethylene

Pyrolysis/Mass Spectrometry (PETREX Collector # 4 - First Wire Analysis)

Figure 5 is the mass spectrum obtained during the Py/MS analysis of PETREX Collector # 4 - First Wire Analysis. The information presented in Table I indicated a total of 86 mass spectral fragments are present in PETREX Collector # 4 - First Wire Analysis having a total ion count of 670,019 of which an ion count of only 98,984 is associated with mass fragments greater than mass 45. The maximum peak greater than mass 45 is the fragment at mass 56 which has an ion count of 14,176. A small fragment at mass 207 is due to column bleed. The Py/MS spectrum obtained during this First Wire Analysis shows the presence of very low concentrations of benzene, toluene (benzene, methyl-), tetrachloroethylene (PCE), and trichlorofluoromethane (F-11).

Figure 5. Py/MS spectrum obtained from Wire # 4 indicating the presence of benzene, toluene, tetrachloroethylene, and trichlorofluoromethane

RESULTS OF PETREX PYROLYSIS/GAS CHROMATOGRAPHY/MASS SPECTROMETRY ANALYSIS OF SECOND WIRES

The utilization of pyrolysis/gas chromatography/mass spectrometry (Py/GC/MS) analysis further enhances the identification of many volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs) using gas chromatographic procedures to permit separate elution of many unknown chemical species, the avoiding the presence of interfering fragments during the mass spectrometric analysis of homologous compounds, isomers which often tend to coelute.

Quality Control Program

System Blank - No-Wire/Py/GC/MS Analysis

Figure 6 is the computer reconstructed mass chromatogram obtained during a Systems Blank - No Wire GC/MS run. Inspection of Figure 6 showed there were two peaks observed above the acceptable noise level:

Peak # 1 Scan 13 - Retention Time (R.T.) = 3.36 min.

Peak # 2 Scan 625 - R.T. = 12.95 min.

Peak # 1 has been tentatively identified as Silane, difluorodimethyl-, M.W. 96; $C_2H_6F_2Si$ during the computer comparison of the spectra obtained during the No-Wire Analysis and the mass spectra present in the National Institute of Standards and Technology (NIST) Library of Mass Spectra. (See Figure 7) Peak # 2 has been tentatively identified as hexamethylcyclotrisiloxane, M.W. 222; $C_6H_{18}O_3Si_3$. (See Figure 8)

Figure 6 is the computer reconstructed mass chromatogram obtained during a Systems Blank - No-Wire GC/MS run

It should be noted that the chemical species observed during the analysis of the No-Wire Systems Blank are usually associated with column bleed and do not interfere with the analysis of environmental samples.

Figure 7. Mass spectrum used in the identification of the unknown chemical species No-Wire Py/GC/MS analysis R.T. = R.T. 3.36 minutes

Table II contains information pertaining to the analysis and tentative identification of the unknowns attributed to column bleed which eluted at 3.36 minutes and 12.95 minutes respectively during the Py/GC/MS/GC/MS No-Wire Standard run. Examination of the data contained within Table II indicated that difluoro-silane had an ion count of only 2,472 while the Hexamethylcyclotrisiloxane had an ion count of 33,960.

TABLE II
MASS SPECTRAL DATA PERTAINING TO UNKNOWN CHEMICAL SPECIES
BELIEVED TO BE RELEASED AS COLUMN BLEED
(Nonwire Blank)

<u>Peak Number</u>	<u>Retention Time Minute</u>	<u>Scan Number</u>	<u>Tentative Identification</u>	<u>Probability % Hit (/)</u>	<u>Ion Count</u>
1	3.36	1	Silane, difluoro-	67 (1/2)	2,472
2	12.95	625	Hexamethylcyclotrisiloxane	93-74 (2/5)	33,960

Figure 8. Mass spectrum used in the identification of the unknown chemical species No-Wire Py/GC/MS analysis R.T. = 12.95 minutes

2 of 2

Special Quality Control Procedures

The development of the PETREX Wire Sensor has provided the capability for the use of a passive soil gas, groundwater, and surface water characterization which permits the assessment of many VOCs in the low parts per billion (ppb) to high parts per trillion (ppt) range under a wide variety of conditions. The nickel Curie-point alloy used for the majority of NERI's environmental surveys has a Curie-point of 358 °C. Other Curie-point alloys permit selection of Curie points ranging from approximately 100 °C. to more than 650 °C. The adhesive used to adhere the sorbent to the wire is nonorganic and does not produce degradation products which can influence the determination of VOCs or SVOCs during field surveys.

Three principal sources of potential interactions which may affect the results obtained during PETREX Surveys are: (1) thermal degradation of VOCs or SVOCs which may produce chemical species which, if special procedures are not employed, may effect the interpretation of analytical results, (2) the presence of low-level volatile chemical species which may be adsorbed to the sorbent during transfer of the sensor from its container to the crystal in which it is introduced into the pyrolysis/analytical system, and (3) the presence of adsorbed volatile species on the sorbent which have not been removed during the manufacture of the sensor and the activation of the sorbent. In order to minimize these potentially adverse factors special quality control procedures are used as will be described below.

Wire Blank - Analysis of Unexposed Wire

A nonexposed wire was introduced into the Py/GC/MS system and analyzed using identical procedures employed in the analysis of PETREX Wires utilized in the field survey. Figure 9 is the computer reconstructed mass chromatogram obtained during the analysis of the nonexposed wire. Table III contains information obtained during the analysis of this nonexposed wire.

Figure 9. Computer reconstructed mass chromatogram obtained during the Py/GC/MS analysis of the nonexposed PETREX wire

TABLE III

**CHEMICAL SPECIES IDENTIFIED AS BEING PRESENT DURING THE PY/GC/MS
ANALYSIS OF THE NONEXPOSED PETREX WIRE**

<u>Peak Number</u>	<u>Retention Time Minute</u>	<u>Scan Number</u>	<u>Tentative Identification</u>	<u>Probability % Hit (/)</u>	<u>Ion Count</u>
1	3.67	33	Butene, isomers	60-31 (5/5)	719
2	3.91	48	Acetaldehyde	60-60 (3/3)	2,276
3	4.00	54	Acetaldehyde	76-70 (5/5)	3,320*
4	5.16	128	Ethanol	76-70 (5/5)	869
5	5.71	163	2-Propanone	78-70 (5/5)	1,771
6	6.10	188	Acetonitrile	70-70 (4/5)	1,959*
7	7.17	256	Not Identified		619**
8	9.49	404	Benzene	76-52 (4/5)	3,593
9	25.28	1411	1,3,5,7-Tetraazatri cyclo-[3.3.1.13.7] decane	78 (1/1)	1,346

* The presence of atmospheric components Argon, Nitrogen, Oxygen, and Carbon dioxide may effect the tentative identification of these compounds without the use of standards.

** Most probably hexane

Background Chemical Species

Table IV contains a listing of chemical species commonly observed during the Py/GC/MS analysis of PETREX Wires as well as several additional chemical species are often observed during the operation of the gas chromatographic/mass spectrometer systems in low concentrations which are attributed to column bleed. The Py/GC/MS data obtained from the NODA Travel Blank and the 4 PETREX Collectors should be compared with the information pertaining to sensor and analytical system background chemical species.

TABLE IV
CHEMICAL SPECIES COMMONLY OBSERVED AS BACKGROUND DURING THE
PY/GC/MS ANALYSIS OF PETREX WIRES

<u>Peak Number</u>	<u>Retention Time Minute</u>	<u>Scan Number</u>	<u>Tentative Identification</u>	<u>Probability % Hit (/)</u>	<u>Ion Count</u>
1	3.68	36	Butene, isomers	76-52 (5/5)	808
2	4.01	57	Acetaldehyde	60 (1/1)	2,602*
3	5.34	142	Not Identified		1,420
4	6.11	191	Acetonitrile	76-70 (4/5)	3,865*
5	8.23	326	Not Identified		1,576
6	8.32	332	Not Identified		3,829
7	13.00	630	Not Identified		1,371
8	13.11	637	Not Identified		1,073
9	13.47	660	Ethene, tetrachloro	87-36 (5/5)	695
10	25.33	1416	1,3,5,7-Tetraazatri cyclo-[3.3.1.13.7] decane	70 (1/1)	718

* The presence of atmospheric components argon, nitrogen, oxygen, and carbon dioxide may effect the tentative identification of these compounds without the use of standards.

The review of the compounds identified on the nonexposed wire standard together with the values of the ion counts for the most intense mass fragment > than 45 a.m.u. clearly indicates that with the possible exception of benzene (a common and almost ubiquitous chemical species found in indoor air at very low levels, the chemical species detected during the thermal desorption/GC/MS analysis are not attributed to environmental pollutants on the NODA site. Table IV contains a summation of the chemical species found to be present as a result of GC column bleed or as low-level background in the computerized GC/MS System.

Travel Blank - Analysis of Unexposed Wire

Travel Blanks are used as part of NERI's Quality Control/Quality Assurance Procedures. A Travel Blank is treated in the identical manner as the PETREX Sensors which are installed in the field except for the fact that the cap sealing the Travel Blank is never opened until it is returned to NERI's laboratory for analysis following shipment from the survey site. Travel Blanks undergo manufacture, storage, shipping, burial in the field, return to NERI's laboratory with the PETREX Collectors which have been exposed in the field, and analyzed with the actual PETREX Collectors utilized in the field survey. If Travel Blanks are contaminated, NERI conducts an analysis of retained PETREX Collectors to determine if the contamination was due to NERI's operational procedure or may be attributed to field handling problems.

Figure 10 is the computer reconstructed mass chromatogram obtained during the analysis of PETREX Travel Blank Collector which was returned from the NODA Site. Table V contains the Py/GC/MS data obtained during the analysis of PETREX PETREX Travel Blank Collector.

Figure 10. Computer reconstructed mass chromatogram obtained during the Py/GC/MS analysis of the PETREX travel blank collector

TABLE V
PY/GC/MS DATA OBTAINED DURING THE ANALYSIS OF THE PETREX
TRAVEL BLANK

<u>Peak Number</u>	<u>Retention Time Minute</u>	<u>Scan Number</u>	<u>Tentative Identification</u>	<u>Probability % Hit (/)</u>	<u>Ion Count</u>
1	3.69	34	Butene, isomers	67-33 (4/5)	716
2	3.94	50	Acetaldehyde	76-70 (5/5)	11,921*
3	5.16	128	Ethanol	70-70 (5/5)	2,691
4	5.72	164	2-Propanone	78-70 (5/5)	1,253
5	7.18	257	Hexane	79-76 (5/5)	1,966
6	8.12	317	Not Identified		1,184
7	8.25	325	Not Identified		2,726
8	9.49	404	Benzene	60-52 (5/5)	1,868
9	12.48	595	Benzene, methyl-	76-70 (3/5)	2,805
10	12.99	627	Hexamethylcyclotrisiloxane	78 (1/2)	5,442
11	14.30	711	Foramide, N,N-dimethyl-	89-38 (2/5)	7,107
12	25.22	1407	1,3,5,7-Tetraazatri cyclo-[3.3.1.13.7]		

* The presence of atmospheric components Argon, Nitrogen, Oxygen, and Carbon dioxide may effect the tentative identification of these compounds without the use of standards.

A review of the chemical species commonly observed as analytical system or PETREX wire background compounds, illustrated in Tables IV and V indicates that of the possible compounds which may be present on the NODA side (benzene and toluene) these two compounds were detected on the PETREX wire at low levels. Benzene was shown to have an ion count of 1,868 which was only 52 percent of that observed during the Py/GC/MS analysis of the control nonexposed wire standard. Toluene was observed during the analysis of the travel blank wire (2,805 ion counts) but was not detected during the Py/GC/MS analysis of the nonexposed wire standard. **It should be noted that both benzene and toluene may be produced as a degradation product of sagebrush which is prevalent on the NODA site.**

Authentic Standards - Benzene, Toluene, and Styrene

Authentic standards (benzene, toluene, and styrene) were utilized in the Py/GC/MS analysis of the Nonwire Systems Blank, the Nonexposed (Virgin) Wire Blank, and the Travel Blank returned from the NODA site. These standards were employed in the assessment of the gas chromatographic Retention Indices (R.T.) as well as in the determination of the probability of match between unknown chemical species adsorbed to the PETREX Wires. The use of authentic standards provides additional information which is necessary to determine the presence or absence of soil gas or ground water contaminants when such contaminants may be present in either very low concentrations or at considerable depth.

Table VI contains a summary of the chemical species detected on a PETREX Wire exposed to the headspace in a tube containing a mixture of authentic standards of benzene, toluene, and styrene each standard being introduced into the headspace tube at a concentration of approximately 250 ppm. The time of the headspace exposure was approximately 20 minutes.

A review of the information presented in Table VI indicated very high ion counts for benzene (166,377), toluene (1,420,296), and styrene (894,149). Other chemical species such as xylene, isomers (3,886), ethynyl-benzene (3,551), 1-methylethyl-benzene (3,044), propyl-benzene (3,278), 1-methylethyenyl-benzene (3,886), benzaldehyde, a degradation product of benzene (75,976), and benzonitrile (2,609) were detected during the Py/GC/MS analysis of the headspace wire; these compounds may be present in the three authentic standards as trace impurities. We have also observed many of these compounds during studies to determine the oxidation and/or degradation of these aromatic standards in previous studies.

TABLE VI
CHEMICAL SPECIES DETECTED DURING PY/GC/MS ANALYSIS OF A PETREX WIRE
IN THE HEADSPACE ABOVE AUTHENTIC MIXED SAMPLES OF BENZENE,
TOLUENE, AND STYRENE

<u>Peak Number</u>	<u>Retention Time Minutes</u>	<u>Scan Number</u>	<u>Tentative Identification</u>	<u>Probability % Hit (/)</u>	<u>Ion Count</u>
1	3.93	49	Acetaldehyde	70-70 (5/5)	2,817*
2	4.01	54	Acetaldehyde	70-70 (5/5)	2,203*
3	5.71	163	2-Propanone	70-70 (5/5)	840*
4	6.11	188	Acetonitrile	70-70 (5/5)	2,529*
5	9.50	404	Benzene	95-78 (5/5)	166,377
6	10.15	446	Not Identified		787
7	12.49	595	Benzene, methyl-	88-86 (5/5)	1,420,295
8	15.13	763	Benzene, ethyl-	83 (1/5)	5,568
9	15.33	776	Xylene, isomers	88-87 (5/5)	3,886
10	15.79	805	Benzene, ethynyl-	70-63 (3/3)	3,551
11	16.08	824	Styrene	97-75 (3/5)	894,149
12	16.77	868	Benzene, (1-methylethyl)-	79-43 (4/5)	3,044
13	17.56	918	Benzene, propyl-	76-76 (3/5)	3,278
14	18.31	966	Benzene, (1-methyl ethenyl)-	96-73 (4/5)	3,886
15	18.63	986	Benzaldehyde	95-76 (7/5)	75,976
16	19.54	1044	Benzonitrile	70-39 (2/5)	2,609
17	19.93	1069	Not Identified		481
18	21.29	1156	Pyridine, ethenyl isomers	26-25 (2/2)	733
19	25.28	1410	1,3,5,7-Tetraazatri cyclo-[3.3.1.13.7] decane	60 (1/1)	886

* The presence of atmospheric components argon, nitrogen, oxygen, and carbon dioxide may effect the tentative identification of these compounds without the use of standards.

Analysis of PETREX Second Wires Returned from the NODA Site Using Py/GC/MS

PETREX Sample # 1 - Second Wire Py/GC/MS Analysis

Figure 11 is the computer reconstructed mass chromatograph obtained during the Py/GC/MS analysis of the PETREX Sample # 1 - Second Wire Collector which was returned from the NODA Site. Table VII contains the Py/GC/MS data obtained during the analysis of Sample # 1 - Second Wire.

Figure 11. Computer reconstructed mass chromatogram obtained during the Py/GC/MS analysis of PETREX Sample # 1 - Second Wire

TABLE VII
PY/GC/MS DATA OBTAINED DURING THE ANALYSIS OF PETREX SAMPLE # 1
SECOND WIRE

<u>Peak Number</u>	<u>Retention Time Minute</u>	<u>Scan Number</u>	<u>Tentative Identification</u>	<u>Probability % Hit (/)</u>	<u>Ion Count</u>
1	3.68	33	Butene, isomers	70-37 (4/5)	990
2	4.02	55	Acetaldehyde	70-60 (5/5)	2,469*
3	5.75	165	2-Propanone	70-70 (5/5)	496
4	6.13	189	Acetonitrile	76-70 (4/5)	6,250*
5	8.27	326	Not Identified		2,026
6	13.01	628	Not Identified		1,668
7	15.14	764	Benzene, ethyl	52 (1/1)	1,744
8	16.12	826	C ₈ H ₈ , MW = 104		1,452
9	25.34	1414	1,3,5,7-Tetraazatri cyclo-[3.3.1.1 ³ .7] decane	60 (1/1)	243

The review of information presented previously within this report, including the results of Py/MS, the systems blanks, the analysis of the non exposed wire, the analysis of the NODA travel blank, and the analysis of the authentic samples strongly indicate that the presence of benzene and toluene, is very low and if actually present most probable exists as a low-level degradation product of sagebrush, or is present at great depth in small concentrations.

PETREX Sample # 2 - Second Wire Py/GC/MS Analysis

Figure 12 is the computer reconstructed mass chromatograph obtained during the Py/GC/MS analysis of the PETREX Sample # 2 - Second Wire Collector which was returned from the NODA Site. Table VIII contains the Py/GC/MS data obtained during the analysis of Sample # 2 - Second Wire.

Figure 12. Computer reconstructed mass chromatogram obtained during the Py/GC/MS analysis of PETREX Sample # 2 - Second Wire

TABLE VIII

PY/GC/MS DATA OBTAINED DURING THE ANALYSIS OF PETREX SAMPLE # 2
SECOND WIRE

Peak Number	Retention Time Minute	Scan Number	Tentative Identification	Probability % Hit (/)	Ion Count
1	3.68	36	Butene, isomers	76-52 (5/5)	808
2	4.01	57	Acetaldehyde	60 (1/1)	2,602*
3	5.34	142	Not Identified		1,420
4	6.11	191	Acetonitrile	76-70 (4/5)	3,865*
5	8.23	326	Not Identified		1,576
6	8.32	332	Not Identified		3,829
7	13.00	630	Not Identified		1,371
8	13.11	637	Not Identified		1,073
9	13.47	660	Ethene, tetrachloro	87-36 (5/5)	695
10	25.33	1416	1,3,5,7-Tetraazatri cyclo-[3.3.1.13.7] decane	70 (1/1)	718

The presence of tetrachloroethylene (PCE) on the PETREX Wire, reported during the Py/MS analysis of the PETREX Sample # 2 - First Wire, has been confirmed by the Py/GC/MS analysis of PETREX Sample #2 - Second Wire, however, the observed ion count of 695 definitely confirms that PCE is present at an extremely low level.

PETREX Sample # 3 - Second Wire Py/GC/MS Analysis

Figure 13 is the computer reconstructed mass chromatogram obtained during the Py/GC/MS analysis of the PETREX Sample # 2 - Second Wire Collector which was returned from the NODA Site. Table IX contains the Py/GC/MS data obtained during the analysis of Sample # 3 - Second Wire.

Figure 13. Computer reconstructed mass chromatogram obtained during the Py/GC/Manalysis of PETREX Sample # 3 - Second Wire

TABLE IX

**PY/GC/MS DATA OBTAINED DURING THE ANALYSIS OF PETREX SAMPLE #3
SECOND WIRE**

<u>Peak Number</u>	<u>Retention Time Minute</u>	<u>Scan Number</u>	<u>Tentative Identification</u>	<u>Probability % Hit (/)</u>	<u>Ion Count</u>
1	3.66	30	Butene, isomers	60-31 (4/5)	637
2	5.33	136	Not Identified		1,191
3	6.09	185	Acetonitrile	76-70 (4/5)	3,643*
4	8.23	321	Not Identified		1,234
5	9.48	401	Benzene	76-37 (5/5)	2,225
6	13.00	625	Not Identified		1,747
7	25.31	1410	1,3,5,7-Tetraazatri cyclo-[3.3.1.13.7] decane	70 (1/1)	540

* The presence of atmospheric components argon, nitrogen, oxygen, and carbon dioxide may effect the tentative identification of these compounds without the use of standards.

The presence of tetrachloroethylene (PCE) on the PETREX Wire, reported during the Py/MS analysis of the PETREX Sample # 3 - First Wire, has not been confirmed by the Py/GC/MS analysis of PETREX Sample # 3. The Py/MS analysis of PETREX Sample # 3 - First wire did indicate that only a trace concentration of PCE was present, the concentration of PCE present is most likely to be below the detection limit.

PETREX Sample # 4 - Second Wire Py/GC/MS Analysis

Figure 14 is the computer reconstructed mass chromatograph obtained during the Py/GC/MS analysis of the PETREX Sample # 4 - Second Wire Collector which was returned from the NODA Site. Table X contains the Py/GC/MS data obtained during the analysis of Sample # 4 - Second Wire.

Figure 14. Computer reconstructed mass chromatogram obtained during the Py/GC/MS analysis of PETREX Sample # 4 - Second Wire

TABLE X

PY/GC/MS DATA OBTAINED DURING THE ANALYSIS OF PETREX SAMPLE # 4
SECOND WIRE

Peak Number	Retention Time Minute	Scan Number	Tentative Identification	Probability % Hit (/)	Ion Count
1	3.58	27	Butene, isomers	52-31 (5/5)	853
2	3.92	49	Acetaldehyde	70-60 (4/4)	169*
3	5.26	134	Not Identified		1,900
4	5.65	159	2-Propanone	70-70 (5/5)	1,875
5	6.02	183	Acetonitrile	78-70 (4/5)	4,378*
6	7.11	252	Hexane	76-60 (5/5)	1,133
7	8.20	322	Not Identified		1,674
8	9.02	374	Cyclohexane	94-88 (5/5)	3,595
9	9.44	401	Benzene	87-63 (4/5)	4,638
10	9.62	412	C ₆ H ₁₀ MW = 82		1,055
11	12.45	593	Benzene, methyl	87-86 (3/5)	3,245
12	12.97	626	Not Identified		2,485
13	25.34	1,414	1,3,5,7-Tetraazatri cyclo-[3.3.1.1.3.7] decane	70 (1/1)	1,094

* The presence of atmospheric components argon, nitrogen, oxygen, and carbon dioxide may effect the tentative identification of these compounds without the use of standards.

A review of the information presented in Figure 14 and Table X indicates that the presence of benzene and toluene, as predicted by the Py/MS analysis of PETREX Sample # 4 - First Wire Analysis, is most likely correct, however, the concentrations of these pollutants as indicated by the ion counts Benzene (4,638) and toluene (3,245) would be expected to be very low. The presence of tetrachloroethylene (PCE) on the PETREX Wire, reported during the Py/MS analysis of the PETREX Sample # 3 - First Wire, has not been confirmed by the Py/GC/MS analysis of PETREX Sample # 3. The Py/MS analysis of PETREX Sample # 3 - First wire did indicate that only a trace concentration of PCE was present, the concentration of PCE present is most likely to be below the detection limit.

Summary and Conclusions

A very limited PETREX Soil Gas Reconnaissance Survey was conducted on the NODA site. This survey was a very limited demonstration survey utilizing only 5 PETREX Collectors. It is possible that benzene and toluene exist on the NODA site, however, based on our very limited reconnaissance survey, it is not possible to determine whether the suspected aromatic compounds benzene and toluene exist as a result of decomposition of terpenes present in sagebrush or whether they may be present resulting from the disposal of solvents used on the NODA site. We believe that there may be very low levels of PCE and trichlorofluoromethane (F-11) on the NODA site. It would be necessary to conduct a follow-up study using a grid of appropriate size and design to provide a more definite assessment of possible NODA site contamination. We would, as part of a second phase study recommend analysis of soil and water samples to confirm the results obtained in our preliminary and limited study of this site.

Very truly yours,

Irving N. Einhorn
President and Chief Executive Officer

cc: J. Viellenave
J. Henrie
M. Hatheway
L.C. Yang

Appendix E

Dual Band Thermal Infrared Measurements—Lawrence Livermore National Laboratory Report

To: Jim Ferguson
From: Mike Carter
Subject: Draft of IR imaging report

Nov 19, 1992

Jim

I am sending you a draft of our report on our IR imaging work at INEL during September of 1992. In preparing this draft it is clear that we have a significant amount of image analysis left to do. After our initial looks at the data turned up little of real interest, we began concentrating on understanding the spectroscopic data taken with the FTIR spectrometer. It is not clear we yet know exactly how to interpret the FTIR data but we are making significant progress. We will turn some of our attention to the image analysis in order to complete this report.

Please forward a copy of this report to Buck Sissons for a review. Please inform Buck that we did receive prints of our photographs this week. These prints are necessary for us to make a complete analysis of the IR images.

I will attempt to keep in touch with Buck and keep him informed of our progress. I apologize for our lack of attention to your schedule. I hope we will make significant progress in generating a final report in the next few weeks.

Best Regards

A handwritten signature in black ink, appearing to be 'Mike Carter', with a long horizontal stroke extending to the right.

Mike Carter

**Draft of Preliminary Report on
Thermal Infrared Measurements
at the
Idaho National Engineering Laboratory**

Michael R. Carter, Charles L. Bennett, and David J. Fields

November 1992

**Lawrence Livermore National Laboratory
P. O. Box 808, L-43
Livermore, Ca 94550**

Abstract

In this report we describe the results of our infrared imaging measurements at the Idaho National Engineering Laboratory. We also lay out a theoretical basis for the expected magnitude of the thermal signature of buried objects. We consider which surface and subsurface variables and conditions are relevant in producing "noise" in infrared images. We discuss the conditions under which buried objects can be detected in principle. We present our field observations of the signals and backgrounds observed with our infrared imaging system. We also present spectral measurements of various regions within these images. We finally present our conclusions on the utility of infrared imaging for the detection of buried waste at the INEL. We do find flaws in an earlier Martin Marietta analysis of the detectability of buried objects by their infrared signature. We rather agree with the conclusions stated in a summary by Josten¹ of the Martin Marietta results specifically:

"Preliminary theoretical calculations made by EG&G Idaho indicate that objects buried below 1 to 2 ft will be undetectable by a thermal system because they will be beyond the penetration depth of solar radiation. These predictions differ substantially from those made by Martin Marietta and included in their final report. At the INEL, the thermal system exhibited no capabilities for detecting buried waste. Thermal anomalies detected at the SDA, SL-1, and the Cold Test Pit buried waste areas were all associated with ground surface features such as topography, vegetation, or soil texture. Due to the lack of promising results for detecting buried waste, no further field demonstrations are warranted. Future work should focus on establishing a sound theoretical basis for detection of buried waste."

Background

As part of the effort to clean up the weapons complex, the Buried Waste Integrated Demonstration (BWID) was initiated by the Office of Technology Development (OTD) within the Environmental Restoration and Waste Management (ERWM) branch of the Department of Energy (DOE). The lead laboratory for this project was chosen to be the Idaho National Engineering Laboratory (INEL). In order to evaluate possible advanced technologies, the IBS (Identification of Buried Structures, Aerial Surveillance and Analysis of Buried Waste) project was initiated in August, 1991. Some of the alternatives considered for the identification of buried ordnance and waste (and their limitations) were:

1. Use of historical records (incomplete or inaccurate).
2. Ground-penetrating-radar (inaccurate depth and identification).
3. Shallow seismic reflection (little reflection from objects near surface, shapes of objects are inaccurately determined)
4. Ground-contact magnetic/electromagnetic reconnaissance (pit geometries were indeterminate).
5. Core boring and sampling (invasive, generates waste, exposure hazard to personnel).
6. Excavation (identification of wastes post-excavation is of no use in guiding the excavation, nor is it helpful in determining which wastes are best treated in situ)
7. Manual survey with metal detector (explosive hazard to personnel)
8. No action. (planning for remediation becomes guesswork).

Two helicopter-mounted technologies were chosen for study by the IBS project. One was a thermal infrared imaging system fielded by Martin Marietta. The second was an EM system fielded by EBASCO. The initial Long-Range plan for the IBS was described in a 1991 report by Williams². An evaluation of the results of the two systems demonstrated was published¹ in March, 1992. The conclusion of the evaluation of the thermal imaging system was summarized by Josten¹.

"Due to the lack of promising results for detecting buried waste, no further field demonstrations are warranted. Future work should focus on establishing a sound theoretical basis for detection of buried waste."

Introduction

A highly precise measurement of the infrared light emitted from a natural scene reveals a great deal of complexity. There is a large degree of temporal periodicity, with strong peaks in the power spectrum corresponding to the daily and annual variations. There is a large degree of spatial variation, with differences produced by topography, biogenic activity, moisture content, and variations in thermal absorptivity and emissivity. Some variations are transient and localized, while other variations are transient and diffuse, or steady and localized, or steady and diffuse. All of this structure is obvious on consideration of the images produced by an infrared camera. The image of a natural scene in the infrared often contains so many features of the structures present, that the image strongly resembles a black and white image produced by a visible light camera, as the differences in light intensity in the visible, which produce the contrast in a visible light image, are often highly correlated with the differences in the light intensity in the infrared.

One prominent exception to the correlation of infrared and visible images is produced by thermal effects. Pressing ones palm against a wall leaves a residual image readily discernable in the infrared for about a minute after the hand is removed. This sensitivity to thermal effects makes possible the detection of shallowly buried structures, since the response to the day/night heating cycle is different for buried objects than for surface objects. More deeply buried, larger structures can be observed, in principle, by their response to the summer/winter heating cycle. The sensitivity to the buried object decreases exponentially with depth of burial, however, so that only relatively shallow objects can possibly be detected with this technique.

Mathematical Details

The differential equation which describes heat conduction is

$$\nabla \cdot (-\kappa \nabla T) = -c\rho \frac{\partial T}{\partial t} + h \quad (1)$$

where T is the temperature, κ is the thermal conductivity, c is the specific heat, ρ is the density, and h is the density of heat production. It will be assumed initially that horizontal variations are negligible compared to variations with depth z , so that the diffusion equation becomes one dimensional. Henceforth, primes

denote derivatives with respect to z , while dots denote derivatives with respect to time t . The one dimensional diffusion equation is

$$T'' + \frac{\kappa'}{\kappa} T' - \frac{1}{\alpha} \dot{T} = - \frac{h}{\kappa}, \quad (2)$$

where α is the thermal diffusivity. If the thermal conductivity is independent of depth this equation becomes

$$T'' - \frac{1}{\alpha} \dot{T} = - \frac{h}{\kappa}. \quad (3)$$

The temperature distribution $T(z,t)$ is a linear combination of a steady state component and a fluctuating component. The local curvature of the steady state component is determined by the local heat production. For the fluctuating component, the time dependence of the density of heat production is presumed negligible, and the diffusion equation becomes homogeneous. The depth profile of the fluctuating component at time t is related to the previous time history of the surface temperature by the expression

$$T(z,t) = \int_0^\infty T(0,t-t') g(z,t') dt', \quad (4)$$

where the green function g is explicitly

$$g(z,t) = \frac{z}{\sqrt{4\pi\alpha t}} \exp\left(-\frac{z^2}{4\alpha t}\right). \quad (5)$$

For periodically varying components in general, the diffusion equation reduces to the Helmholtz equation

$$T'' + \frac{i\omega}{\alpha} T = 0, \quad (6)$$

and the temperature profile with depth is given by

$$T(z,t) = T(0) \exp\left(-z \sqrt{\frac{\omega}{2\alpha}} (1-i) - i\omega t\right). \quad (7)$$

The attenuation length δ is $\sqrt{2\alpha/\omega}$. (In this equation, and henceforth, it is to be understood that physical values of

observables correspond to the real part of the complex expressions.) For a medium with a uniform thermal diffusivity, the attenuation length for the daily cycle is $\sqrt{365} \approx 19$ times shorter than the attenuation length for the annual cycle. Because of the strong attenuation with depth of high frequency components in the time dependence, the time dependence of the subterranean temperature eventually becomes nearly a perfect sine wave regardless of the surface weather conditions.

The behavior of the temperature profile of eqn. 7 is identical to the attenuation of electromagnetic waves impinging on a good conductor. The "skin depth" for attenuation of electromagnetic waves is a direct analog of the attenuation length δ .

Subterranean measurements³ of the temperature vs time are shown in Figures 1 to 6. It can easily be seen that the high frequency components in the time dependence of the surface temperature are rapidly attenuated with depth, so that at the deepest measured depth, the data is visually indistinguishable from a pure sine wave. From these data, it is apparent that the approximation of nearly constant thermal diffusivity is reasonable for the region studied. Furthermore, a value of the attenuation length for the annual variation, $\delta_a = 1.75$ m, can be accurately determined. The value of the attenuation length for the daily variation for this material is $\delta_d = 9$ cm.

Using these values for the attenuation lengths for daily and annual periods, idealized calculations of the temperature profile vs depth at various times of the year have been plotted in Figures 6 and 7. In these calculations all frequencies except the daily and annual variation have been ignored. Modulations for frequencies at the daily rate and above will be completely undetectable below approximately 50 cm.

We have noted that the calculations by the Martin Marietta group on the sensitivity of the thermal signature for buried waste were erroneous⁴. They seem to have confused the daily and the annual cycles. In effect, they assumed that the daily temperature cycle was able to penetrate as far as the annual cycle. One of their "conclusions" about buried waste was that "A positive contrast (i.e. hot target) can be expected from 8 pm to 8 am, A negative contrast (i.e. cold target) can be expected from noon to 6 pm". This is only true for objects buried no more than a few inches deep, and is simply wrong for objects buried more than a foot deep.

For the problem of buried waste, we are primarily concerned with objects buried below 50 cm. Only the annual cycle of

temperature variations will penetrate significantly to these depths, and we will limit the following analysis to this single frequency component. For the idealized case of an abrupt change in the thermal diffusivity, as might be produced by a large array of barrels, buried a uniform distance underground, a "reflection coefficient" may be defined as the ratio of the amplitude of the thermal wave which is reflected upwards to the amplitude of the incident thermal wave which is travelling into the earth. Let δ' be the attenuation length in the buried layer, and δ be the attenuation length for the surface layer. The value of the reflection coefficient is

$$R = \frac{\delta' - \delta}{\delta' + \delta} \exp(-2D \frac{1+i}{\delta}) \quad (8)$$

There are two simple limiting cases, either the buried medium is a very good thermal conductor, so that δ' is very large, and the factor in front of the exponential becomes unity, or the buried medium is a very good thermal insulator, so that δ' vanishes, and the prefactor equals -1. In the event that the thermal diffusivity contrast is small, the reflection coefficient also becomes small.

One characteristic signature of a buried layer is that there be a reflected thermal wave appearing at the surface with a phase lag with respect to the driving thermal wave produced by surface heating. The magnitude of the out of phase component, due to the subterranean reflection, relative to the driving component is given by

$$\frac{\delta' - \delta}{\delta' + \delta} \exp(-\frac{2D}{\delta}) \sin(\frac{2D}{\delta}) \quad (9)$$

For the measured diffusivity which fit the temperature measurements displayed in Figures 1 to 6, the magnitude of this perturbation vs D , the depth of the buried object, has been graphed in Figure 9. Under the most optimistic circumstances the surface temperature perturbation is at most a few degrees centigrade. For a more realistic case, consisting of a layer with a diffusivity contrast of only 50%, the prefactor in equation 9 is 1/3, and only 1° of temperature contrast is likely. The greatest contrast between the in phase component and the out of phase component appears at either

the autumnal or vernal equinox, while the minimum contrast appears at the summer or winter solstice.

Unfortunately, the mere observation of a temperature difference between two regions at a given time of year is not sufficient to identify a buried structure. The average temperature T_0 for a given location is a function of the absorptivity at the short wavelengths corresponding to the dominant insolation wavelengths for the solar spectrum, and the emissivity in the infrared range of wavelengths near $10\text{ }\mu\text{m}$, corresponding to the dominant region of ground emission. The average temperature is also sensitive to other heating and cooling mechanisms. Topography and vegetation affect the influence of wind cooling, for example. Non-radiative factors can also have both daily and annual cycles. These competing factors are all involved in determining the baseline temperature upon which the daily and annual variations take place. Furthermore, even if a given patch of ground has the same temperature as its surroundings, but has a somewhat higher short wavelength emissivity, or if it has a somewhat lower long wavelength emissivity, this region will appear "hotter" to an ir camera.

Spatial Resolution Limitations

The maximum possible spatial resolution for infrared images of buried objects is fundamentally limited by the spreading of the reflected heat wave as it propagates from the buried object to the surface. The best resolution possible in principle is limited to approximately the burial depth of the object. For buried waste at INEL, with depths reachable only by the annual wave, there is an additional limit on the spatial resolution of the images of buried structures. This blurring is caused by the tendency for a temperature contrast to come to equilibrium with its surroundings. A hot spot tends to warm its surroundings, both by conduction (through the ground) and convection (through the air). Over the period of a year, the efficacy of this equilibration can be highly variable, and dependent on the local topography, vegetation, and weather. In summary, for an array of barrels buried 1 to 2 meters underground in a pit, for example, it would be extremely unlikely to be able to locate the pit boundary to better than a couple of meters.

LLNL IR imaging measurements

Infrared imaging studies were conducted by Lawrence Livermore National Laboratory personnel at the Idaho National Engineering Laboratory (INEL) in September of 1992. Measurements were made at the United States Geological Service (USGS) test site

just north of the Subsurface Disposal Area (SDA). Measurements were also made at the location of the thermal anomaly found by Martin Marietta at the Naval Ordnance Disposal Area (NODA). Diagrams showing the USGS site and the NODA site are located in Figures 10 and 11. The intent of these measurements was to determine the effectiveness of dual-band thermal imaging in detecting deeply buried objects.

Measurements were made at several times throughout the nighttime hours. Measurements were made at the USGS site on two separate nights. Measurements were made at the NODA site on only one night. Visible photographs were taken during the daytime hours in order to help document the locations of fiducials, vegetation and other man-made objects located within the IR images.

Instrumentation

Infrared imaging measurements were made using a dual band Thermovision®880 infrared scanner system from Agema Infrared Systems. The scanner system employs two IR scanners, one uses an InSb detector for the short-wavelength IR region, the other uses a HgCdTe detector for the long-wavelength IR region. The short wavelength scanner is sensitive to photons with wavelength between 2 and 5.6 μm . The long-wavelength scanner is sensitive from 8 to 12 μm . The analog signals from the scanners are digitized with 12 bit precision. The minimum detectable temperature difference of the Thermovision®880 scanners is 0.2° C.

The infrared scanners were mounted on a bucket truck and lifted approximately 55 feet above the ground. The cameras were mounted on a remotely operated pan and tilt mechanism and the camera control computer was remotely operated. This allowed complete operation from a mobile vehicle and eliminated the need to place a person in the bucket truck to control the camera system. Most measurements were made with a 20 degree field of view lens. This corresponds to a 19 foot field of view on the ground (assuming a vertical view). This limited field of view necessitates the use of periodic spatial fiducials to allow one to create a mosaic of several images. The spatial fiducials also permit accurate coregistration of the two IR bands and allow the use of algorithms to separate the image's thermal and emissivity components. This technique will be described in detail below.

Dual-Band Infrared Imaging technique

The data analysis of the dual-band infrared images⁷ exploits a property of Plank's law that applies for temperatures near room temperature. The intensity of infrared emission at wavelength λ as a function of temperature, T , can be approximated by a power law

$$I(\lambda) = k \epsilon T^{(50/\lambda)} \quad (10)$$

where k is a constant, ϵ is the emissivity, T is the temperature in degrees K, and λ is the wavelength in microns. This power law fit to the intensity is shown in Figures 12 and 13 for 5 micron and 10 micron infrared radiation.

Using the ratio of the measured infrared radiation in the two IR bands we can compute the following:

$$R = I(5)/I(10) = [\epsilon(5)/\epsilon(10)] * T^5 \quad (11)$$

For a blackbody, $\epsilon(5) = \epsilon(10)$, and this ratio is proportional to the temperature to the fifth power.

$$R \sim T^5 \quad (12)$$

We can also calculate the emissivity ratio by computing the following

$$I(10)^2/I(5) = \epsilon(10)^2 * T^{10}/(\epsilon(5) * T^{10}) = \epsilon(10)^2/\epsilon(5) \quad (13)$$

We typically compute normalized ratios to obtain the temperature and emissivity ratio maps:

$$T = \ln[(S/s)/(L/l)] \quad (14)$$

and

$$\epsilon = \ln[(S/s)/(2L/l)] \quad (15)$$

where S is the intensity in the 5 micron band, s is the average intensity in the 5 micron band, L is the intensity in the 10 micron band and l is the average intensity in the 10 micron band.

In the case of a blackbody or graybody, the unprocessed thermal image and the temperature ratio map will look alike. In the case of a blackbody or graybody the emissivity ratio will be a

constant. In other cases, these ratios can help between actual temperature related features within an image and emissivity variation within the image.

Image analysis

A typical long wavelength IR image from the NODA site is shown in Figure 14. This image was taken at 3:00 am on September 15, 1992. This image shows the edge of the thermal anomaly (a compacted gravel pad) and the berm surrounding the pad. The infrared camera calibration indicates a temperature difference of over 2.5 degrees K between the gravel pad and the berm. This is in close agreement with the 2.1 degree measurement made by a temperature probe inserted just below the surface of the soils. Figure 15 shows the effective temperature ratio as given in equation (14). There is indeed a measurable temperature difference between the gravel pad and the surrounding berm area. Figure 16 shows the calculated emissivity ratio as given in equation (15). Note that the emissivity does not vary appreciable between the two areas. The average emissivity can be taken as a constant to better than 2%. We discuss below the effects of non-thermal emission from the gravel pad.

The source of the temperature difference between the gravel pad and the surrounding soil has yet to be identified. It is clear that there is a significantly different density, heat capacity, thermal conductivity and thermal diffusivity between the two soils. It also seems likely that the surface reflectivity, in the visible region of the spectrum, is quite different. There are small differences in emissivity in the infrared region of the spectrum. These differences are discussed below. Any of these effects can cause a difference in temperature between the two areas. The exact cause of the temperature may be able to be determined by accurate knowledge of the time history of the temperatures. The temperature difference can also be modeled if independent measurements of the critical quantities can be made.

We are continuing to analyze the time history of the IR measurements at the NODA site in hopes of gaining insight to the cause of the temperature difference. There is no evidence of buried structures below the gravel pad.

Figure 17 shows a composite view of the USGS site. Note the location of the caisson at the far left of the image. Although analysis of this data is incomplete at this time, we have yet to find a significant variation in surface temperature attributable to the buried objects within this view. We believe this is consistent with our analytic analysis presented above. A complete statistical analysis of the images has yet to be done.

Spectroscopic Study at the NODA site

One of the findings of the Martin Marietta surveys of the NODA site at INEL, was the identification of a large area whose infrared signature differed from most of the rest of the landscape. It was speculated that this area could perhaps be the site of a chemical spill.

Spectrally resolved infrared measurements were made at the NODA site using a portable MIDAC FTIR spectrometer (model M2400). This FTIR spectrometer has an acceptance angle of approximately two degree and is therefore not an effective imaging instrument. The spectral resolution of this FTIR spectrometer is variable. All measurements at the NODA site were made with a resolution of 0.5 cm^{-1} . The FTIR spectrometer was mounted on a tripod at a height of about 4 feet above the ground. This corresponds to a field of view of about 2 to 3 inches on the ground. This spot size is small enough to isolate the thermal emission of the major features found at the NODA site including: the gravel pad, the surrounding soil, and vegetation.

In order to help identify the reason for the different appearance in the infrared of the NODA site, spectroscopic measurements from a selection of regions near the NODA site were measured. Figure 18 displays a spectrum taken of a sample of dirt dug up from the soil just outside the "infrared anomalous" region. The shape of this spectrum is generally consistent with a black body distribution corresponding to the ambient temperature, once the instrumental efficiency is factored out. Figure 19 displays a comparison of the spectra of in situ dirt outside the "ir anomalous" region with in situ gravel within the "ir anomalous" region. In the raw spectra, it can be seen that there is more structure in the gravel spectrum than in the dirt spectrum. In order to see this structure more clearly, both of the spectra in Figure 19 were divided by a reference spectrum, and the results plotted in Figure 20. The interval plotted in Figure 20 corresponds approximately to the 8 to $12 \mu\text{m}$ "water window" of air transmission. The NODA gravel clearly shows a lower emittance feature in the vicinity of 1100 cm^{-1} wavenumbers, i.e. about $9 \mu\text{m}$. It is known that all silicates have a strong band at just about this wavelength (see reference 8, which contains a study of nearly 50 silicates). It is very likely that the gravel in the NODA site, which is readily visible to the naked eye, is a silicate material. The emissivity contrast apparent in the Martin Marietta images of the NODA site can be simply attributed to a difference in the nature of the surface material. The variation in

radiance can not be solely attributed to a temperature difference of the two materials, since there is so much spectral structure.

Figure Captions

1. The observed variations of temperature (°C) at depths of 0.6 m and 0.9 m during an annual cycle are compared with a sine wave.
2. The observed variations of temperature (°C) at depths of 1.5 m and 2.1 m during an annual cycle are compared with a sine wave.
3. The observed variations of temperature (°C) at depths of 2.7 m and 3.4 m during an annual cycle are compared with a sine wave.
4. The sine wave parameters are plotted vs depth in the ground. The frequency is precisely consistent with the annual cycle. The average temperature is nearly independent of depth.
5. The temperature amplitude is plotted vs depth, and compared with a linear fit.
6. The phase of the sinusoidal temperature variation is plotted vs depth, and compared with an exponentially decreasing fit.
7. Schematic illustration of the depth dependence of the temperature for summer vs winter.
8. Schematic illustration of the depth dependence of the temperature at the autumnal equinox.
9. Plot of the "out of phase" surface temperature perturbation due to a perfectly conducting buried layer as a function of the depth of the layer.
10. Schematic of the USGS test site at INEL.
11. Schematic of the NODA site at INEL.
12. Plot of the IR intensity at 5 microns vs temperature using Planck's Law and the approximate power law $I(\lambda) = k \epsilon T^{(50/\lambda)}$
13. Plot of the IR intensity at 10 microns vs temperature using Planck's Law and the approximate power law $I(\lambda) = k \epsilon T^{(50/\lambda)}$
14. An image from the NODA site showing the Gravel pad on the right and the surrounding dirt on the left. The four bright spots are spatial fiducials used for image registration.
15. The calculated relative temperature using the dual band IR analysis technique. This is an image from the NODA site (the same location as Figure 14).
16. The calculated emissivity ratio from the NODA site showing both the gravel pad and the berm.
17. A composite view of the USGS test site at INEL. These images were taken in the 8-12 micron range.

18. A raw FTIR emission spectrum of dirt sample from near the NODA site. Instrumental response has not been factored out of this data. The units on the abscissa are inverse centimeters or wavenumbers.
19. A raw FTIR emission spectrum of in situ NODA gravel is compared with in situ NODA dirt spectrum.
20. A relative emission spectrum of in situ NODA gravel is compared with a relative emission spectrum from NODA dirt. Notice the decrease in emissivity near 1100 wavenumbers.

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Fig 4

Fig 7

Fig 1

INEL - USGS Site - Schematic

INEL - NODA - Schematic

Fig 12

Fig 13

NOAA Raw image 8-12 μ m

Temperature map

Emissivity Ratio

E-34

Arbitrary Y

NODA01

Res= 4cm-1

11/13/92 15:47

Noda dirt

Gain=0.16 . F 18

E-35

NODAFD3

Res= 4cm-1

11/13/92 15:41

Fig 1f

E-36

NODAFLD0

Res= 4cm-1

11/13/92 15:36

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Appendix F

Geophysical Surveys

Appendix F

Geophysical Surveys

Since the TIR anomaly could be the result of unrecorded waste disposal activities, a geophysical survey for buried waste was conducted. The magnetometer in the vertical gradient mode is sensitive to the presence of magnetic objects. Thus, the magnetometer was selected to detect the presence of buried objects, which typically contain high levels of ferrous metals. Results of the survey on a 2-m grid spacing and the boundary of the gravel pad estimated from plane table mapping are shown on Figure F-1. The largest perturbation to the vertical gradient was caused by the large iron gate and gate posts at the southeast corner of the gravel pad (lower right corner of Figure 4 in the main section of this report). The areas of immediately west of the center of the gravel pad with closed contours (Figure F-1) indicated the possibility that a buried metallic object was present. A more dense grid spacing of 0.2 m was used to survey the area with closed contours and the results (Figure F-2) indicate that the cause of the anomaly was shallow (less than 1-m) and had a low magnetic mass (indicated by the small amplitude of the vertical gradient anomaly). The results of these surveys demonstrated that the gravel pad and the access areas to the gravel pad were free of large, buried magnetic objects such as steel drums, canisters, and boxes typically found at INEL solid waste sites.

The site was surveyed using a Model EM-31 ground conductivity meter (Geonics, Mississauga, Ontario, Canada), which was operated in quadrature mode to detect the presence of any large electrical conductors and to map the subsurface conductance. A 2-m grid spacing was used. The EM-31 results were contoured (Figure 4) and indicate that the gravel pad and the soils beneath the gravel pad have a relatively low and relatively uniform electrical conductivity. Therefore, no large electrical conductors exist beneath the gravel pad. The absence of large, electrical conductors indicates that the site was not used for the disposal of inorganic liquids such as acidic, basic, or saline solutions.

Ground-penetrating radar (GPR) (operated at 100 and 500 kHz), was used to further characterize the area of the magnetic anomaly found in the magnetometer survey. GPR responds to changes in dielectric properties of soil layers or buried materials. The GPR did not respond consistently over the area of the magnetic anomaly. The lack of a consistent response indicates that either no object was present or that the radar signal did not penetrate the soils very well. Based on previous use of the gravel pad area (see Appendix A) and the general lack of response from the geophysical surveys, the site was deemed to be free of buried metal objects typical of buried waste sites at the INEL. Therefore, no evidence was found indicating that the gravel pad was created during undocumented waste disposal activities.

Figure F-1. Vertical gradient in earth's magnetic field (gamma/m) over the gravel pad at NODA. Small areas near the center of the pad (areas with closed contours) are the magnetic anomaly. The region in the southeast corner of the figure shows the effects of an iron gate and gate posts at the entrance to the NODA site. Data were obtained in a 2-m grid.

Figure F-2. Vertical gradient in earth's magnetic field over the magnetic anomaly shown in Figure F-1. The coordinates correspond to the local grid used in Figure F-1. Data were obtained on a 0.2-m grid.

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