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SOIL CORE CHARACTERIZATION STRATEGY AT DNAPL SITES SUBJECTED TO STRONG THERMAL OR CHEMICAL REMEDIATION

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ABSTRACT: At Launch Complex 34 (LC34), Cape Canaveral Air Station, Florida, high concentrations of trichloroethylene (TCE) exist in groundwater and free phase (DNAPL) has been detected in the subsurface. Characterizing or monitoring sites that are contaminated with volatile organic compounds (VOCs) such as TCE is often challenging because of the difficulties associated in minimizing VOC loss during soil sample handling and collection. New difficulties in post-demonstration soil sampling were encountered due to (1) the residual strong oxidant remaining in the soil cores of the oxidation remediation plot; and (2) the high temperatures (50-95ºC) in the thermal remediation plot that persisted for several months after the remediation had been completed. To evaluate the efficiency of the soil collection and sampling method in recovering VOCs, hot soil cores were brought to the surface and spiked with a surrogate compound, 1,1,1-trichloroethane (1,1,1-TCA). The results show that between 84 and 113% of TCA in the soil was recovered using field procedures designed to minimize VOC loss while protecting personnel handling the hot soil cores. The results also indicate that any VOC loss occurring during cooling of the soil core is minimal and within the acceptable limitations of the field sampling protocol.

INTRODUCTION

Dense nonaqueous-phase liquid (DNAPL) contamination presents a persistent environmental problem at many federal and private facilities. At Launch Complex 34 (LC 34), Cape Canaveral Air Station, Florida, high concentrations of trichloroethylene (TCE) exist in groundwater and free phase (DNAPL) has been detected in the subsurface. The Interagency DNAPL Consortium (IDC), a consortium consisting of U.S. Department of Energy (DOE), U.S. Department of Defense (DoD), U.S. Environmental Protection Agency (EPA), and the National Aeronautic and Space Administration (NASA), has been assessing several innovative DNAPL remediation technologies, including those that use thermal or chemical oxidation treatment. Post-demonstration characterization was conducted to verify the effectiveness of these innovative technologies, which were demonstrated in separate test plots in the DNAPL source zone.

Characterizing or monitoring sites that are contaminated with volatile organic compounds (VOCs) such as TCE is often challenging because of the difficulties associated in minimizing VOC loss during soil sample handling and collection. At LC 34, new difficulties in post-demonstration soil sampling were encountered due to (1) the residual strong oxidant remaining in soil cores of the chemical oxidation remediation plot; and (2) the high temperatures (50-95ºC) in the thermal remediation plots that persisted for several months after the remediation had been completed. Field procedures for soil core handling and sampling were designed to take into account the safety issues posed by the strong oxidant (potassium permanganate) used at LC34 in the chemical

oxidation plot (Battelle, 1999). Field procedures for collecting soil cores and soil samples from the thermal remediation plot were modified in an effort to minimize VOC losses that can occur as a result of contaminant volatilization associated with elevated soil temperature (Battelle, 2001). Because additional consideration must be given to issues such as personnel safety when handling hot soil cores, there is the possibility that increased handling times during soil coring and sample collection may result in an increase in VOC losses. An experiment was conducted using soil samples spiked with a surrogate compound to investigate the effectiveness of the field procedures developed for LC34 in minimizing VOC losses. Because the soil sampling procedures were similar for both the chemical oxidation and thermal remediation strategies, the remainder of this paper focuses on issues associated with the collection and sampling of soil at elevated temperatures.

MATERIALS AND METHODS

Soil cores were collected in a 2-inch diameter, 4-foot long acetate sleeve that was placed tightly inside a 2-inch diameter stainless steel core barrel. The acetate sleeve was immediately capped on both ends with a protective polymer covering. The sleeve was placed in an ice bath to cool the heated core to below ambient groundwater temperatures (approximately 20ºC). The temperature of the soil core was monitored during the cooling process with a meat thermometer that was pushed into one end cap (see Figure 1). Approximately 30 minutes was required to cool each 4-foot long, 2-inch diameter soil core from 50-95ºC to below 20ºC. Upon reaching ambient temperature, the core sleeve was then uncapped and cut open along its length to collect the soil sample for contaminant analysis (see Figure 2).

Soil samples were collected in relatively large quantities (approximately 200 g) along the entire length of the core rather than sampling small aliquots of the soil within

FIGURE 1. A soil core capped and cooling in an ice bath. The thermometer is visible in the end cap.

FIGURE 2. A soil sample being collected from along the length of the core into a bottle containing methanol.

the core, as required by the conventional method (EPA SW5035). This modification is advantageous because the resultant data provide an understanding of the continuous VOC distribution with depth. VOC losses during sampling were further minimized by placing the recovered soil samples directly into bottles containing methanol (approximately 250 mL) and extracting them on site. The extracted methanol was centrifuged and sent to an off-site laboratory for VOC analysis. Soil samples taken from the chemical oxidation plot were handled similarly, although they did not require cooling. The soil sampling and extraction strategy is described in more detail in Gavaskar et al. (2000).

To evaluate the efficiency of the sampling method in recovering VOCs, hot soil cores were extracted from 14 through 24 feet below ground surface and spiked with a surrogate compound, 1,1,1-trichloroethane (1,1,1-TCA). The surrogate was added to the intact soil core by using a 6" needle to inject 25 μL of surrogate into each end of the core for a total of 50 μL of 1,1,1-TCA. In order to evaluate the effect of the cooling period on VOC loss, three soil cores were spiked with TCA prior to cooling in the ice bath and three cores were spiked with TCA after cooling in the ice bath. In the pre-cooling test, the surrogate was injected as described above and the core barrels were subsequently capped and placed in the ice bath for the 30 minutes of cooling time required to bring the soil core to below 20°C. A thermometer was inserted through the cap to monitor the temperature of the soil core.

In the post-cooling test, the soil cores were injected with TCA after the soil core had been cooled in the ice bath to below 20°C. After cooling, the caps on the core barrel were removed and the surrogate compound was injected in the same manner, 25 μL per each end of the core barrel using a 6" syringe. The core was recapped and allowed to equilibrate for a few minutes before it was opened and samples were collected. Only for the purpose of the surrogate recovery tests, the entire contents of the sampling sleeve were collected and extracted on site with methanol. The soil: methanol ratio was kept approximately the same as during the regular soil sample collection and extraction. Several (four) aliquots of soil and several (four) bottles of methanol were required to extract the entire contents of the sample sleeve.

Two different capping methods were used during this experiment to evaluate the effectiveness of each cap type. Two of the soil cores were capped using flexible polymer sheets attached to the sleeve with rubber bands. The remaining four soil cores were capped with tight-fitting rigid polymer end caps. One reason that the polymer sheets were preferred over the rigid caps was that the flexible sheets were better positioned to handle any contraction of the sleeve during cooling.

RESULTS AND DISCUSSION

The results from the surrogate spiking experiment are shown in Table 1. Soil cores 1, 3, and 5 received the surrogate spike prior to cooling in the ice bath. Soil cores 2, 4, and 6 received the surrogate spike after cooling in the ice bath. The results show that between 84 and 113% of the surrogate spike was recovered from the soil cores. Recovery comparison is not expected to be influenced significantly by soil type because all samples were collected from a fine grained to medium fine-grained sand unit. The results also indicate that the timing of the surrogate spike (i.e., pre- or post-cooling) appeared to have only a slight effect on the amount of surrogate recovered. Slightly less surrogate was recovered from the soil cores spiked prior to cooling. This implies that any

losses of TCA in the soil samples spiked prior to cooling are minimal and acceptable, within the limitations of the field sampling protocol. The field sampling protocol was designed to process up to 300 soil samples that were collected over a 3-week period, during each monitoring event.

TABLE 1. Recovery in soil cores spiked with 1,1,1-TCA surrogate

Soil Cores Spiked Prior to Cooling	Capping Method	1,1,1-TCA Recovery (%)	Soil Cores Spiked After Cooling	Capping Method	1,1,1-TCA Recovery (%)
Core 1	Flexible polymer sheet with rubber bands	96.3	Core 2	Flexible polymer sheet with rubber bands	98.7
Core 3	Rigid End Cap	101.0	Core 4	Rigid End Cap	112.6
Core 5	Rigid End Cap	84.3	Core 6	Rigid End Cap	109.6

The capping method (flexible versus rigid cap) did not show any clear differences in the surrogate recoveries. The flexible sheets are easier to use and appear to be sufficient to ensure good target compound recovery.

This experiment demonstrates that the soil core handling procedures developed for use at LC34 were successful in minimizing volatility losses associated with the extreme temperatures of the soil cores. It also shows that collecting and extracting larger aliquots of soil in the field is a good way of characterizing DNAPL source zones.

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