CHAPTER 5

Bench- and Pilot-Scale Studies

5.1. Introduction and Considerations in Determining Testing Approaches.

5.1.1. The goals of a bench- or pilot-study typically are:

a. Feasibility demonstration.

b. Evaluate process design and potentially optimize.

It is therefore important to define the need for a bench or pilot-scale testing and the goals that are to be achieved. Bench-scale testing assesses the treatability of a particular compound or suite of compounds and defines potential chemical reactions (adverse or desired) when heating occurs. Pilot testing typically confirms design parameters in situations where uncertainty exists about treatment of compounds of concern or process performance under site-specific conditions. For large and complex sites, the use of bench- or pilot-scale testing can supplement the modeling and assist the engineer or scientist in determining if ISTR is an appropriate means to remediate a site.

5.1.2. Bench-scale tests usually consist of column studies. Column tests gauge the effectiveness of the ISTR technology on specific contaminants existing at the site. ISTR has been demonstrated to be effective for most VOCs. However, if the chemicals of concern are not VOCs, or there is a mixture of chemicals of concern, column studies should be performed to evaluate the feasibility of ISTR technologies. Column studies refer to packing a column with site soil and site contaminants, applying a representative ISTR technology, and measuring effluent concentrations as a function of the time. The test results usually cannot be directly scaled up to the full-scale ISTR system unless the site lithology consists of homogeneous isotropic soils.

5.1.3. Because pilot testing is expensive, it can be justified if uncertainties arise that (1) are critical to the success of the project and (2) can be answered by the pilot. Therefore, the pilot must be carefully designed to obtain critical data and not simply to satisfy curiosity. Pilot-scale tests are conducted at the site, the location of which is determined by the goals of the pilot test and site-specific constraints. The pilot should be of sufficient size to encompass a significant portion of the site variability, or the results from the pilot test may not be applicable to the rest of the site. A pilot test is usually designed so that the pilot test system can be incorporated into the full-scale system should a full-scale application of the ISTR be merited. This phased approach can significantly speed the overall remediation process. The size of the equipment installed for the pilot test should be carefully considered if such a phased approach is to be used so that, if the test is successful, the rest of the remediation can be cost-effectively conducted.

5.1.4. A pilot test should achieve the following goals:

5.1.4.1. *Mass Removal*. A pilot test may be used to demonstrate that the ISTR technology can remove contaminant mass at sufficient rates and has the potential to achieve the remedial goals. It should be kept in mind that the most readily extracted fraction of the contaminant mass would be removed by advection no matter what ISTR technology is chosen for the pilot test. The removal rate is expected to decline sharply after a period of time when diffusion-limited mass transfer ensues (except in the case of thermal conductive heating). When designing the pilot test, the test should be long enough to accurately capture the long-term contaminant removal rate.

5.1.4.2. *Radius of Influence*. The radius of influence of each steam injection well, ERH electrode, or thermal conduction well can be calculated theoretically. However, a pilot test should be designed to provide the vadose and saturated zone responses to the application of heating. The well-field layout in the full-scale system can be adjusted accordingly.

5.1.4.3. *Subsurface Characteristics*. A pilot test can provide information on the nature and variability of site-specific subsurface parameters, such as soil permeability, hydraulic conductivity, soil moisture retention, and contaminant distribution.

5.1.4.4. *Design Parameters*. A pilot test would provide valuable information on the design parameters, such as the size of the equipment and treatment system for the full-scale application. ISTR pilot tests may be designed to meet the remedial goal at a small area of the site; for this situation, it is relatively simple to scale up the system to remediate the site. The size of the treatment system can be adjusted if inadequacies are discovered during the pilot test.

5.1.4.5. *Cost Estimates*. The cost for full-scale system implementation and operation can be deduced from the pilot test. Some adjustments may need to be made if the well spacing is found inefficient or if the treatment system size has to be scaled up.

5.1.5. Performing a pilot test in an sub-area of the main portion of the plume risks impacts from influx of contamination from surrounding areas during and following treatment. This complicates the interpretation of mass removal rates and the achievable contaminant concentrations. Several published reports on pilot testing of ISTR methods have noted these complications. Also, adjustments made to the pattern of the injection and extraction wells or electrodes to carve out a small section for the pilot may limit the ability of the system to heat the entire area, or may cause the spread of contaminants.

5.1.6. A pilot-test work plan should be prepared before conducting pilot- or bench-scale tests. The work plan is crucial for specifying test objectives, the range of operation conditions, and parameters to be monitored, including the location, methods, and frequency of measurements to be taken. A Site Safety and Health Plan (SSHP) is also required to assure safety of all on-site workers. A schedule showing critical tasks and the various phases of the work should be included. A materials list for necessary equipment and supplies should be prepared. Necessary permits should be obtained for the pilot system installation and discharge streams. Refer to EM 1110-1-4001, Paragraph 4.4, for Work Plan Requirement and Preparation.

5.2. Thermal Conductive Heating.

5.2.1. *Bench-Scale Studies*. Two general types of bench-scale thermal conduction studies can be outlined:

a. Feasibility Demonstration.

b. Process Design Evaluation.

5.2.1.1. *Feasibility Demonstration*. The principal focus of the feasibility demonstration is to confirm that the soil will become decontaminated by TCH and to determine the extent of remediation as a function of temperature and, to a lesser extent, the duration of heating.

5.2.1.1.1. Bench-scale feasibility may be demonstrated by placing a soil sample in a cylindrical metal tube and passing air through the sample while heating the assembly within a muffle furnace. A thermocouple inserted into the soil sample monitors that the soil is heated to the target temperature and then maintained at that temperature for the requisite amount of time (e.g., 48–72 hr), while minimizing thermal overshoot.

5.2.1.1.2. A typical objective is to demonstrate the extent of removal of COCs as a function of target temperatures; therefore, temperatures are selected to cover the range of interest based on the properties of the COCs. For example, 200, 250 and 300°C were selected to test soil contaminated with explosive compounds such as hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX), octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazine (HMX), 2,4,6-trinitrotoluene (TNT), nitroglycerin, and isomers of 2,4- and 2,6-dinitrotoluene (DNT). As the compounds being tested were SVOCs, the soil undergoing testing was homogenized prior to subsamples for thermal testing being collected. Analysis of pre- versus post-treatment samples enabled assessment of the degree of contaminant removal as a function of temperature and treatment time (Baker et al. 2001).

5.2.1.2. *Process Design Evaluation*. The principal focus of the process design evaluation is to design and optimize a cost-effective remediation program. The total remediation program consists of the in situ heating requirements, plus the associated air quality control (AQC) system for the control of well field emissions. In addition to the fundamental demonstration of the decontamination of the soil, additional issues that are addressed during a process design evaluation program are:

a. What contaminants are desorbed from the soil (as opposed to being destroyed in situ) and at what characteristic temperatures?

b. If there are chlorinated organics present in the soil, does the chlorine exit the soil matrix as a chlorinated hydrocarbon or as hydrochloric acid (HCl) vapor? And to what extent is the gaseous HCl neutralized by the buffering capacity of the soils?

c. For the contaminants desorbed from the soil matrix, can the emissions be adsorbed on activated carbon or is a thermal oxidizer or acid gas scrubber required to reduce the emission levels to below air discharge limits?

d. For the contaminants desorbed from the soil matrix, will the hydrocarbons desorb in a manner such that char (i.e., coke) formation in the vacuum extraction wells is a concern? And should supplemental precautions, such as air bleeds, be provided to clean the extraction wells in the event of excessive char deposition?

5.2.1.2.1. In general, the cost of the process design evaluation is greater than a feasibility demonstration, with the principal differences being the additional analytical work and the time required to do two sequential studies. The only circumstance where a feasibility demonstration should be done before the process design evaluation is where the issue of achievement of treatment objectives or the appropriateness of TCH for a given COC is in question. PCBs and dioxins fall within the former category (i.e., contaminants whose treatability with TCH has been well established) and new applications of TCH, such as mercury removal, are an example of the latter.

5.2.1.2.2. To gain insight into the process design issues, several modifications to the standard feasibility demonstration apparatus and testing procedures are instituted:

a. A custom test chamber is utilized with uniform heating and multiple temperature measurements in the soil sample.

b. The purge gas flow rate through the test chamber is controlled at a much lower flow rate.

c. The off-gases pass through a gas cleanup train consisting of a condenser and activated carbon trap. Additional filtering through a packed bed scrubber is used when the contaminants of concern include significant concentrations of chlorinated hydrocarbons.

d. The off-gases are monitored for total hydrocarbon level, either by use of an FID detector or a combustible gas detector.

e. Air sampling and impinger tubes may also be used to examine the loading of a specific COC or its vapor-phase concentration.

f. The test chamber temperatures are charted to detect short-term transients, in addition to data-logged temperature data.

5.2.1.2.3. The data produced during the process design evaluation is analyzed using procedures appropriate for the COCs in the soil matrix. Further, these data are also used to select and design the AQC equipment. As such, each process design evaluation effort and analytical plan is configured for the site-specific requirements of the individual remediation application.

5.2.1.2.4. Data produced during feasibility demonstrations or process design evaluations can provide valuable input parameters for simulation modeling during design.

5.2.2. *Pilot-Scale Studies*. Thermal conduction pilot tests have been performed at several sites, including the Missouri Electric Works Superfund Site in Cape Girardeau, Missouri, (Vinegar et al. 1997) and the BADCAT demonstration at the former Mare Island Naval Shipyard in Vallejo, California. The following paragraphs describe a recommended approach.

5.2.2.1. *Thermal Wells*. Figure 5-1 depicts a typical pilot test in which 13 thermal wells (one central heater-vacuum well and 12 heater-only wells) are installed in a triangular array at a spacing of 1.83 m (6 feet). A wider interwell spacing, such as 2.13m, may be called for at full-scale, but a narrower spacing can save time during the pilot test, with the results being easily scalable to other spacing following the Inverse Square Law. Suitable electrical distribution equipment is required to power the thermal wells.

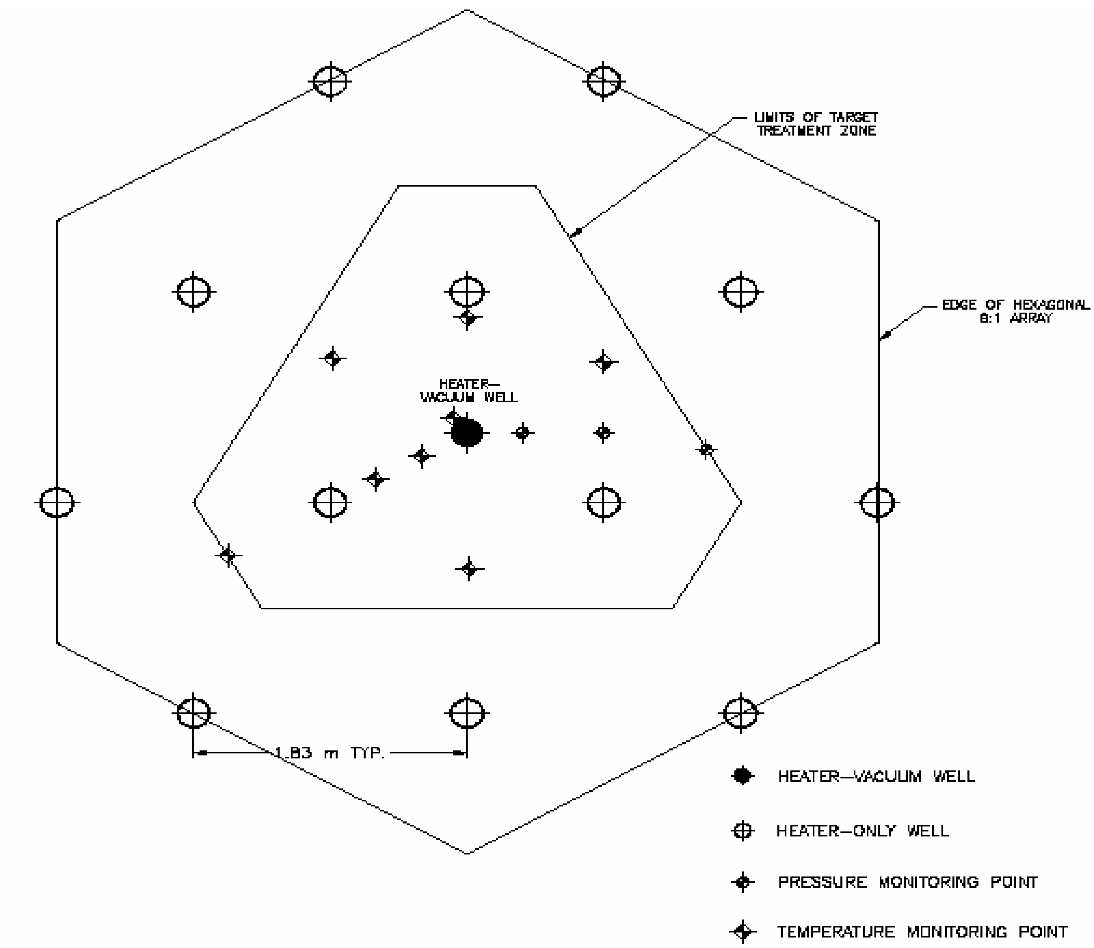

Figure 5-1. Typical Thermal Conduction Pilot-Test Layout.

5.2.2.2. *Monitoring Points*. Several thermocouple arrays (allowing measurement of temperature at various depths) and several discrete soil gas pressure monitoring points are installed to enable tracking of subsurface temperature and pressure gradients between the thermal wells. Figures 5-1 and 5-2 illustrate typical locations of monitoring points relative to the positions of the thermal wells.

5.2.2.3. *Surface Cover*. A surface cover is typically placed over the pilot test area to serve as a vapor barrier, reduce heat-loss, and seal out rainfall (Figure 5-2).

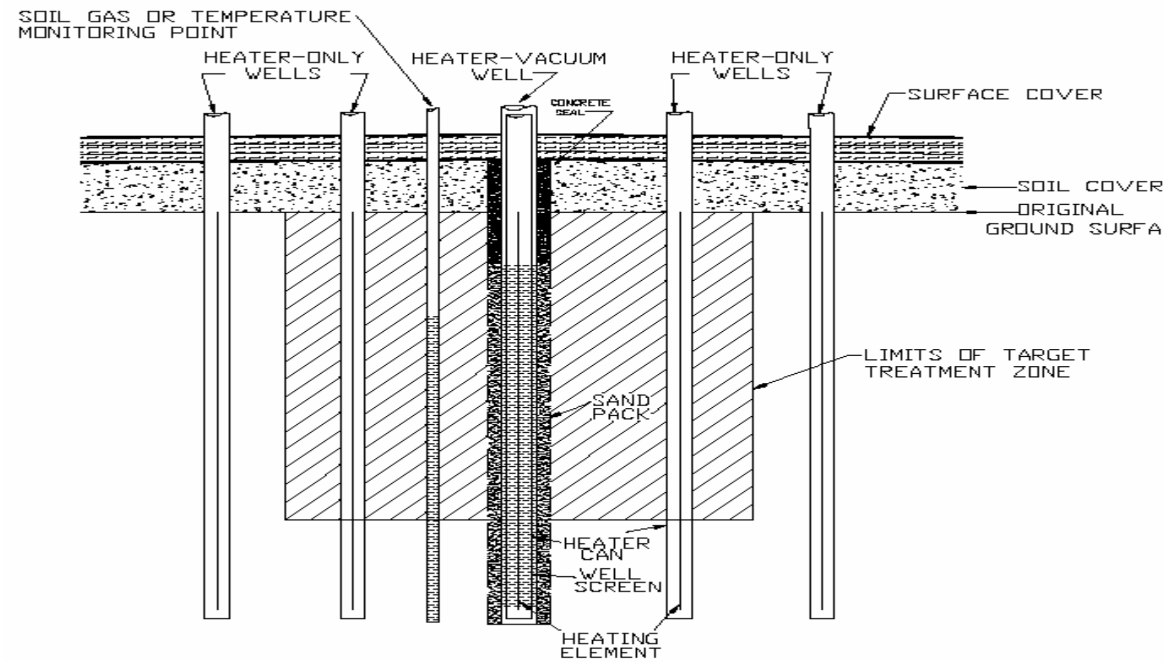

Figure 5-2. Typical Thermal Conduction Pilot-Test Cross-Section (example 1 in text).

5.2.2.4. *Air Quality Control (AQC) System*. The central heater-vacuum well is connected to a vacuum extraction manifold leading to an AQC system. Depending on the COCs, examples of AQC systems are: 1) thermal oxidizer, heat exchanger, scrubber and carbon beds, and 2) carbon bed only. A blower, discharge stack, and air monitoring equipment are also provided (Figure 5-3). A condenser (not depicted in Figure 5-3) is generally used in these systems.

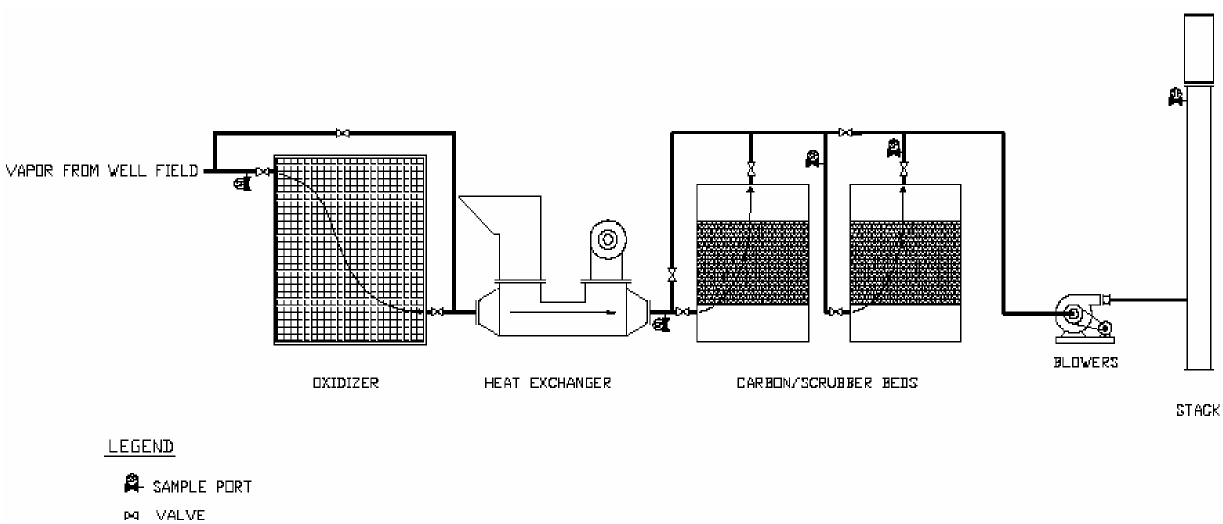

Figure 5-3. Typical Thermal Conduction Pilot-Test Process Diagram.

5.2.2.5. *Operation*. With the AQC system on, the thermal wells are energized, typically for a period of 1–2 months, as is needed to achieve target temperatures in the interwell locations within the Target Treatment Zone (TTZ), depicted in Figures 5-1 and 5-2. Air emissions are monitored to ensure compliance with standards, and to evaluate performance.

5.2.2.6. *Soil Sampling*. Direct-push methods (e.g., hand or power augering; Geo-Probe®) are used to sample soil prior to and following treatment at representative locations within and near the TTZ. Because soils treated using ISTD tend to take weeks or months to fully cool, post-treatment sampling of hot soils is often recommended. Since the core samples are taken from a very hot environment and may be kept in closed barrels that can be cooled far below their original temperature, it is unlikely that significant volatile compounds are lost from even warm cores. One method involves collecting the samples in metal liners, immediately capped upon retrieval, and cooled with ice to ambient temperatures (Gaberell et al. 2002). Once cooled, the liners are opened and subsampled for laboratory analysis of all applicable COCs.

5.2.3. Other items that are commonly examined during field-scale pilot (depending on site) include:

a. Groundwater infiltration/control adequacy/appropriateness.

b. Subsidence.

c. Rate and nature of hydrocarbon off-gassing.

d. Water production rate.

e. Coking.

f. Site-specific thermal conductivity.

5.3. Electrical Resistivity Heating.

5.3.1. *Bench-Scale Studies*. Bench tests are usually not done prior to electrical resistivity heating (ERH) projects. Removal of VOCs through steam distillation is a physical process that is relatively well understood in comparison to the removal processes of most other remediation techniques. However, an ERH bench test may be warranted under the following circumstances:

a. The treatment region consists of landfill debris or other non-soil material.

b. The treatment region includes peat layers or buried wood debris (the effects of extremely high TOC levels are not well known at present).

c. The target VOC is dissolved into a greater mass of oil or other low volatility hydrocarbon (Raoult's Law effects are difficult to predict).

d. For determining attainable concentrations, where significant groundwater flow exists, or to evaluate materials of construction (e.g., to determine corrosion potential).

5.3.1.1 For best results, bench testing should be performed in triplicate. An ERH bench test usually includes the following steps:

a. Pre-test aliquots of soil are analyzed.

b. Soil is packed into a small sealed reactor vessel and heated, usually by placing the reactor in a muffle furnace (for the purpose of bench testing, the method of heating is not important).

c. As the soil is heated, a very small amount of air is injected into the reactor as a carrier gas and the off-gas from the reactor is condensed.

d. When a target condensate production is reached (typically 10 to 15 volume percent of the soil sample), heating is terminated and the reactor is cooled to 4°C and a post-test soil sample is analyzed.

5.3.2. *Pilot Tests*. Pilot tests may be conducted using either three or six phase modes of heating. Typically, pilot tests have been conducted to demonstrate feasibility rather than evaluate a process design or optimization. This is because the process design is relatively straightforward using ERH. Pilot testing typically involves installing a six phase array of electrodes to heat a selected portion of the impacted area to be treated. The major difference between a pilot- and full-scale application is the number of arrays and, hence, the capacity of the power control unit and ancillary treatment equipment.

5.4. <u>Steam Enhanced Extraction</u>. Laboratory treatability studies for steam injection should be done when the contaminants are semi-volatile and, thus, some question exists of the amount of steam or energy that might be needed to achieve the desired residual concentrations of contaminants in the soil, or to estimate the likely residual concentrations for a given number of pore volumes of steam injection. When there is a mixture of contaminants present, it may also be beneficial to determine the physical properties of the non-aqueous phase liquids as a function of temperature. Brief descriptions of the laboratory experiments follow.

5.4.1. *Physical Properties*. Density can be measured as a function of temperature using a method similar to ASTM D1217-93, with a water bath to control the temperature of the sample in the range from ambient temperatures to near steam temperatures. These data can be used to determine if DNAPLs, such as creosote or coal tar, will become LNAPLs as the temperature is increased. Because chlorinated solvents remain denser than water at temperatures below their boiling point, this testing is of less use for chlorinated NAPLs.

5.4.1.1. Viscosity can be measured by a method similar to ASTM D1296, using a jacketed beaker to maintain the sample at the desired temperature and allowing measurements to be made ranging from ambient to steam temperatures. These data demonstrate the increased mobility of the liquid as the temperature is increased. Generally, contaminants such as creosote or coal tar, which have relatively high viscosities at ambient temperatures, will show significant increases in mobility as the temperature is increased. Chlorinated solvents generally have relatively low viscosities at ambient temperatures, and the increased mobility with temperature is not as great. Surface and interfacial tension measurements can also be made using ASTM D971 and jacketed beakers to maintain the samples at the desired temperature. Decreases in surface and interfacial tension with temperature may reduce residual liquid saturations and allow more of the contaminants to be collected as a liquid, and, therefore, more of the high boiling point contaminants may be recovered.

5.4.2. *Steam Injection Experiments*. The laboratory setup for a one-dimensional steam injection experiment is shown in Figure 5-4. Metering pumps are used to deliver a set flow rate (50–100 mL•hour^{-1} for the size soil sample used here) of water to the steam generator. The power input to the steam generator was set to produce steam at a temperature of 150ºC (302ºF). Steam is injected into the top of the soil column to produce a vertical downward flow through the column. Galvanized steel columns, approximately 5 cm (2 inches) in diameter and 15 cm (6 inches) long, which are threaded on both ends, and for which endcaps, can be purchased are convenient for these experiments. Clean sand should be placed in the bottom endpiece of the column to facilitate collection of the effluent steam and recovered contaminants, and the column itself is then filled with approximately 600 to 700 g (1.3 to 1.5 pounds) of contaminated soil from the site. Soil from the most contaminated areas of the site should be used. The top endpiece of the column is not packed with soil to help distribute steam evenly to the top of the soil. Thermocouples can be used at the effluent of the steam generator, in the top of the column, at the midpoint of the column, and in the effluent line. The column is wrapped with heater tape and then covered with insulation to ensure that the entire column remains at steam temperatures. Rubber-coated heater tape, 2.5 cm- (1 inch) wide and 1.22 m (4 feet) long, has been used to wrap the column. The heater tape is plugged into a variable autotransformer to control the heat input to the column.

5.4.2.1. Column effluent goes through a heat exchanger before being collected in a sample bottle immersed in an ice bath. For most cases, two to four pore volumes of steam will be adequate to recover most of the contaminants. Effluent sample containers should be changed approximately every half pore volume so that approximately how rapidly the contaminants are recovered as a function of the pore volumes of steam injected can be determined. However, when determining the sample size, the size needed for the chemical analysis to be performed should be considered. For some semi-volatile contaminants that are present in the initial soil at high concentrations, there may be the potential for them to condense and plug the effluent line. Care should be taken to monitor the sample collection and the pressure in the column to determine if this is happening. If volatile contaminants are being recovered, collection of the effluent vapors may be improved by using a solvent, such as dichloromethylene or methanol, in the sample collection bottle to trap the vapors.

5.4.2.2. During the steam injection, it is important to monitor the amount of steam injected versus the amount of effluent collected. As a steam front is established in the column, it will displace in front of it the liquids that were initially present in the soil pores. Thus, the mass of effluent collected will be greater than the mass of water injected as steam owing to the significantly greater volume of steam versus that of water. If the soil was packed wet and the mass of effluent collected is not greater than the mass of water injected during the early stages of the steam injection, then a steam front has not been established in the column and a hot water flood is taking place. Temperature in the column should also be monitored to indicate whether the experiment is a steam flood or a hot water flood. A temperature plateau at 100ºC indicates that evaporation is occurring in the column and thus a steam front is present and expanding. Once the water within the column has evaporated, the temperature may increase owing to heat input from the heater tape. In some cases, the temperature may be above 100ºC, but pressure within the column can cause condensation of the steam and thus a hot water flood. For most types of contaminants, a steam flood is going to recover significantly more of the contaminants than a hot water flood. Thus, it is important to ensure that a steam front is formed in the column to get a valid laboratory test of the steam enhanced extraction technology.

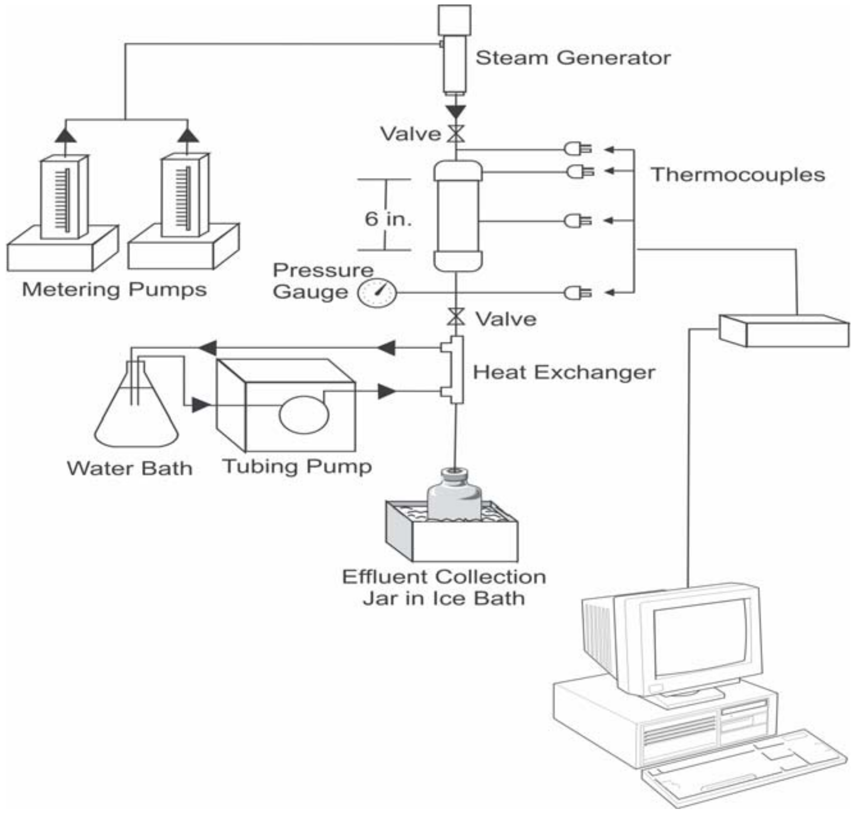

Figure 5-4. Laboratory Setup for One-Dimensional Steam Injection Experiments.

5.4.2.3. After a steam front has been established throughout the column, pressure cycling can be used to increase contaminant recovery rates as a vapor. This is done by closing a valve on the effluent line of the column (while steam injection continues) allowing pressure to build up in the column. The pressure should be monitored by a pressure gauge in the effluent line above the valve. After approximately 70-100 kPa (10-15 psi) of pressure has built up in the column, the valve is opened and the pressure is allowed to dissipate. This process mimics pressure cycling that can be done in the field during steam injection to increase the rate of volatile contaminant recovery.

5.4.2.4. After completion of the steam injection, the column should be taken apart and the soil divided into two or three samples (for example, top, middle, and bottom of the column) for analysis. A sample of the initial soil should also be analyzed.

5.4.2.5. It may be desirable to know the groundwater concentrations of contaminants in equilibrium with the residual contaminants adsorbed onto the soil after a steam injection. If so, water can be added to the column after the steam injection is complete and allowed to equilibrate with the steamed soil for a period of time. Twenty-four hour contact periods have been used in the past; however, that time may not be adequate for full equilibrium between the adsorbed contaminants and the water. For comparison, an initial leachate sample should also be obtained before steam injection is started. After the equilibration period, the water is drained from the column and analyzed separately.

5.4.3. *Pilot Tests*. For SEE, pilot tests may be carried out:

a. To confirm design parameters, such as well spacing, injection pressures and rates, pumping rates, etc.

b. To assess feasibility of technology with respect to site conditions or contaminant-e.g. applications in bedrock where the technology is less proven, determine feasibility with high boiling point compounds, soil stability or stratification issues.

c. Where there is uncertainty in the operation of the treatment equipment in dealing with the ranges of concentrations that may be encountered.

Most of the considerations regarding the approach to pilot tests discussed in Paragraph 5.1 are applicable to SEE.