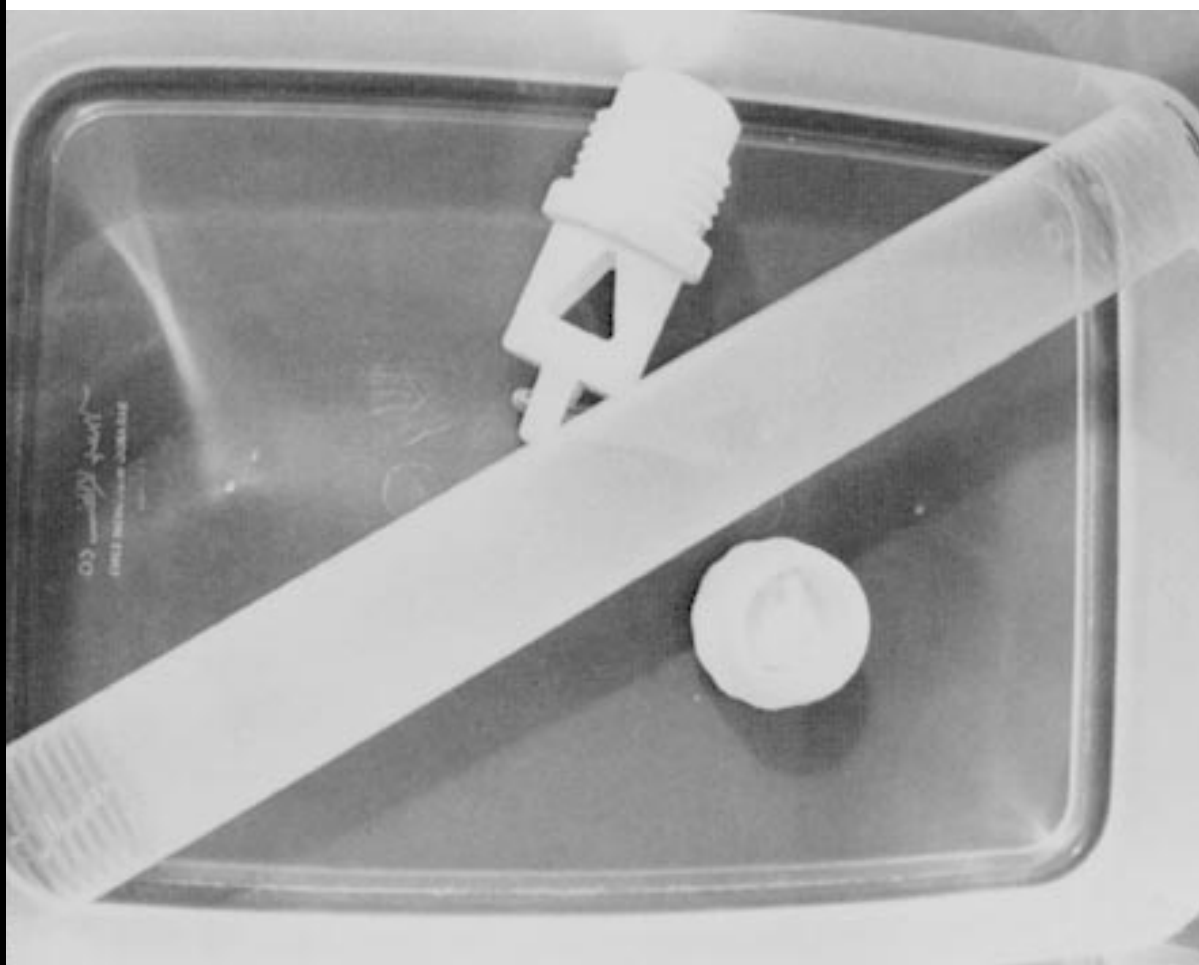# A Literature Review on Decontaminating Groundwater Sampling Devices
## Organic Pollutants
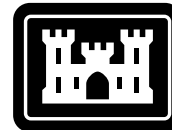
Louise V. Parker

July 1995

***Abstract***

Current protocols for decontaminating devices used to sample groundwater for organic contaminants are reviewed. Most of the methods given by regulatory agencies provide little scientific evidence that justify the recommended protocols. In addition, only a few studies that actually compared various decontamination protocols could be found in the open literature, and those studies were limited in their scope. Various approaches for decontamination and criteria that are important in determining how effectively a surface could be decontaminated are discussed.

*Cover: Single check-valve bailer, disassembled in a cleaning bath. (Photo by Peter Keene.)*

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**US Army Corps of Engineers**

Cold Regions Research & Engineering Laboratory

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**PREFACE**

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# A Literature Review on Decontaminating Groundwater Sampling Devices
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LOUISE V. PARKER

## INTRODUCTION

Dedicating a sampling device to a particular well reduces the potential of cross contamination from other wells. However, many sampling devices currently are not dedicated, and decontamination methods are used to reduce the possibility of cross contamination. In addition, some current regulatory guidance (e.g., U.S. EPA 1992) recommends that dedicated sampling devices that are routinely stored outside the well should be cleaned or decontaminated prior to use. Typically, decontamination procedures for sampling devices range from simply washing with clean (high-quality) water to complex cleaning involving acid and solvent rinses. It is the purpose of this paper to review the literature on decontamination of groundwater sampling devices that are used for sampling organic contaminants.

The type of sampling device and surface(s) to be decontaminated and the physical characteristics of the organic contaminant, such as its solubility in water, its volatility, and its propensity to adsorb on or absorb into materials used in the sampling device, will determine how readily decontamination can occur. Contact time and the degree of initial contamination on the surface are also critical factors. Cleaning a surface that has been exposed to trace-level organic contaminants is much easier than cleaning a surface exposed to neat (pure product) hydrocarbons or organic solvents. Other types of contaminants such as grease and oil may also affect removal. Generally, nonpermeable surfaces such as metals will be more readily decontaminated than permeable surfaces, because desorption of absorbed contaminants may be a factor with permeable surfaces. Temperature also has an effect on these processes, with sorption and desorption slower in the cold.

## SORPTION AND DESORPTION OF ORGANIC CONTAMINANTS

Several studies have shown that nonpermeable surfaces (e.g., glass, stainless steel) do not sorb most organic contaminants. Specifically, two studies (Gillham and O'Hannesin 1990, Parker et al. 1990) have shown that, under conditions where there should not be any losses due to biological activity, there were no losses of a suite of organic contaminants from aqueous solutions exposed to stainless steel surfaces. (The contaminants in these studies all had log $K_{ow}$ values less than 4.) Reynolds et al. (1990) also found no loss of volatile organic compounds (VOCs) (log $K_{ow}$ < 4) from aqueous solutions exposed to glass surfaces. However, more hydrophobic contaminants, such as polychlorinated biphenyls (PCBs), chlorinated pesticides, and polyaromatic hydrocarbons, most likely would be adsorbed by these surfaces. Thus, decontamination of nonpermeable surfaces such as metals and glass would involve removing only those highly hydrophobic contaminants that have adsorbed to the surface (i.e., no absorbed analytes) and any surface contaminants contained in any residual film (either wet or dry) that is left on the surface when the sampling device is removed from the well.

In contrast, several studies (Barcelona et al. 1985, Reynolds and Gillham 1985, Gillham and O'Hannesin 1990, Parker et al. 1990) have shown that permeable materials, such as polymeric well casings and tubings used in sampling pumps, sorb substantial quantities of some organic contaminants from aqueous solutions. All these researchers agree that diffusion within the polymer matrix has occurred. In several studies (Barcelona et al. 1985, Gillham and O'Hannesin 1990, Ranney and Parker, in prep.), researchers have noted that generally the less rigid the material, the more

sorptive it is. Flexible tubings such as those used to pump water samples to the surface have been shown to be very highly sorptive (Barcelona et al. 1985, Reynolds and Gillham 1985, Gillham and O'Hannesin 1990, Ranney and Parker, in prep.). For example, Barcelona et al. (1985) found flexible polyvinyl chloride (PVC) and silicone rubber tubing sorbed 80% of the chloroform in an aqueous solution (100 ppb) in 1 hour. Gillham and O'Hannesin (1990) compared sorption of low (~1) ppm levels of six aromatic hydrocarbons (benzene, toluene, ethylbenzene, and o-, m- and p-xylene) by rigid and flexible PVC. After 10 minutes, losses of these analytes ranged from 0 to 2% for solutions exposed to rigid PVC vs. 35 to 50% for those exposed to flexible PVC tubing.

Leggett and Parker (1994) were able to successfully model (predict) equilibrium sorption of organic solutes by polytetrafluoroethylene (PTFE, one type of Teflon) and rigid PVC based upon meaningful physical/chemical interaction parameters of the organic contaminants. They used multiparameter linear solvation energy relationships (LSERs) to describe the equilibrium partitioning. Partitioning was related to the compound's basicity, acidity, polarity/polarizability, and molecular volume. These models proved to be more accurate than octanol–water partition coefficients in correlating the experimental partitioning data. This approach may also prove useful for predicting how readily a surface can be decontaminated or if desorption of analytes that have been absorbed within the polymer matrix could be rapid and extensive enough to affect analyte concentrations.

Thus, for permeable materials, decontamination may involve more than removing surface contaminants, depending upon the kinetics of sorption and desorption, contact times, and contact volumes. An example of this can be seen when Devlin (1987) tried to decontaminate tubings made of polyethylene and an unspecified fluoropolymer (Teflon) that had been exposed to ppb levels of a suite of VOCs. Devlin found it was harder to decontaminate polyethylene tubing than the fluoropolymer tubing. At least 5 L of deionized water was required to reduce the concentrations of these analytes in the leachates from the fluoropolymer tubing to below 1 ppb, and the leachates from the polyethylene tubing still contained concentrations greater than 1 ppb of several analytes after 8 L flush. One explanation for these results may be that polyethylene is more sorptive of organic solutes than Teflon tubing (Barcelona et

al. 1985, Devlin 1987, Ranney and Parker, in prep.) and thus decontamination (desorption) takes longer.

Understanding the kinetics of desorption of organic contaminants from permeable materials is critical to determining the potential for effective decontamination. Unfortunately, only a few studies (Miller 1982, Barcelona et al. 1985, Parker et al. 1990) have examined desorption of organic contaminants from polymeric materials. Specifically, Miller (1982) looked for desorption of several volatile organic analytes from polyethylene, polypropylene, and (rigid) PVC. Parker et al. (1990) looked for desorption of a suite of organic contaminants (VOCs, nitroaromatics, and a nitramine) from (rigid) PVC and PTFE. Although neither of these studies followed the kinetics of this process, they both found that the amount desorbed did not always parallel the amount sorbed; e.g., Parker et al. (1990) found that although p- and m-dichlorobenzene were sorbed the most rapidly and to the greatest extent by PTFE, trichloroethylene (TCE) and trans-1,2-dichloroethylene appeared to be desorbed the most rapidly (since they were desorbed to the greatest extent after 3 days). (A biocide was added to prevent any losses due to biological activity in this study.) For PVC, the amount desorbed tended to correlate with the amount sorbed. However for PTFE, it appeared that smaller molecules were more easily or rapidly desorbed than larger molecules, perhaps because diffusion is more rapid for smaller molecules.

Barcelona et al. (1985) compared desorption of chloroform from five flexible tubing materials (polypropylene, polyethylene, PVC, silicone rubber, and PTFE). They found that with all the polymers, 80 to 90% of the desorption they observed occurred almost immediately (i.e., within the first 5 to 10 minutes) although, the amount desorbed constituted less than 10% of the total chloroform sorbed. After 5 minutes, polypropylene and polyethylene desorbed more of the total chloroform sorbed (on a percentage basis) even though the silicone rubber and PVC tubings had sorbed more chloroform more rapidly.

According to several researchers (Brusseau and Rao 1989, Di Cesare and Smith 1994), soil organic matter can be envisioned as a three-dimensional matrix of randomly oriented polymer chains with a relatively open, flexible structure. An apparently similar pattern of desorption of nonionic organic compounds from natural soils has also been reported in kinetic studies (Pavlostathis and

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Jaglal 1991). Desorption consists of an initial rapid phase followed by a continual slower phase. As an example, Di Cesare and Smith (1994) reported that Pavlostathis and Mathavan (1992) found approximately 80 to 90% of the TCE sorbed by a silty clay soil was desorbed in the first day. They also found that the longer the soil was exposed to TCE (2.5, 5.5, or 15.5 months), the more TCE appeared to remain bound (i.e., the less desorption occurred).

As with the permeable surfaces, there has been very little study of desorption of organic contaminants from impermeable surfaces. Parker et al. (1990) found no desorption of any organic analytes from stainless steel that had been exposed to ppm levels of a suite of organic solutes (VOCs, nitroaromatics, and a nitramine) for six weeks. These results were expected since there had been no sorption of these analytes by the stainless steel surfaces during the study. More hydrophobic contaminants would most likely be adsorbed by these surfaces and could then desorb.

## DECONTAMINATION METHODS

A surface can be decontaminated either by physically removing the contaminants or by chemically neutralizing them. Methods that physically remove contaminants include several aqueous cleaning methods, such as detergent washes and high-pressure cleaning methods. High-pressure cleaning methods involve either using a high-pressure washer or steam cleaning. For organics with poor solubility in water, cleaning or rinsing with less polar organic solvents is a viable alternative. There are a number of other methods that physically remove contaminants (e.g., using abrasives, contact heating, burning, hot gases, microwave heating, etc.). However, many of these methods are destructive and do not appear practical or necessary for most routine field cleaning and thus are not included in this discussion. Most currently used methods for decontaminating groundwater sampling devices utilize some combination of aqueous and nonaqueous cleaning methods. Although methods that physically remove contaminants from surfaces can be effective for decontaminating impermeable surfaces, permeable materials can still contain considerable quantities of contaminants within the internal structure, and this can reduce the efficacy of these methods. Another problem with these methods is that the contaminants are often still contained in the waste

product and thus the waste must also be considered potentially hazardous. Chemical neutralization methods can be more effective in reducing contamination and are discussed briefly.

**Aqueous cleaning methods**

*Detergent cleaning*
There are numerous approaches to aqueous cleaning. Typically, small items can be cleaned by soaking in the cleaning solution (in a bath); cleaning by hand using a cloth, sponge, brush, or similar device; ultrasonic cleaning in a bath; or machine washing. Larger items are typically cleaned in place by circulating the cleaning solution and rinse solutions.

Water acts as a solvent medium for contaminants, depending upon their solubility. Water can also act as a dispersal medium for substances that do not dissolve but can be carried in suspension. An aqueous cleaning solution must first wet the contaminated surface. Wetting means that the liquid solution readily spreads over the entire surface. If wetting does not take place, the liquid beads up and will run off any vertical or inclined surface. When a cleaning solution wets a contaminated surface, the contaminant is separated from the solid surface by the cleaning film. The wetting ability of liquids varies: water has less wetting power than ammonia, benzene, acetone, and most oils (Sandia Laboratories 1969). However, the wetting ability of water can be increased by reducing its surface and interfacial tension; this is done by adding a surface active agent, or surfactant, such as soap or detergent.

For aqueous systems, surfactants are organic compounds that have a two-component molecular structure; one component is hydrophilic and water-soluble and the other is hydrophobic and relatively water insoluble (Di Cesare and Smith 1994). Surfactants are commonly categorized by the ionic character of the hydrophilic component or long chain. The long chain may be negatively charged (anionic), positively charged (cationic), or neutral (nonionic). Amphoteric or ampholytic surfactants are also available; they also ionize in solution but the long chain can carry either a positive or negative charge, depending upon the pH of the solution (Osipow 1962).

A detergent solution removes contaminants from a surface in the following manner:
1) It wets the contaminated surfaces,
2) It removes the contamination by either rolling it up and displacing it from the surface,

or by removing it in layers from the surface, or by removing it in layers by emulsification, or by penetrating the contamination to form inverse micelles, followed by the formation of liquid crystals and solubilization or emulsification,

3) It disrupts the polyvalent cation links and hydrogen bonds by pH control, ion exchange, and sequestrating the polyvalent cation (forming coordination complexes), and

4) It prevents redeposition by maintaining the contaminants in suspension (Osipow 1962).

The concentration of surfactant at which monomers assemble into aggregates (micelles) is termed the critical micelle concentration (cmc) (Di Cesare and Smith 1994). Many organic compounds that are not very soluble in water dissolve to a considerably greater extent in micellar solutions of both ionic and nonionic surfactants (i.e., where the concentration is greater than the cmc); this process is called solubilization (Osipow 1962). Direct evidence that solubilized materials are present within the micelles is provided by X-ray measurements (Osipow 1962). Solubility enhancements of (nonionic) organic compounds at surfactant concentrations below the cmc have only been reported for extremely water-insoluble compounds, e.g., DDT (Di Cesare and Smith 1994).

In general, according to Osipow (1962), surface activity is due to nonmicellar surfactant, and the micelles act as a reservoir for the unassociated surfactant molecules and ions. At concentrations greater than the cmc value, the surface tension of the solution does not decrease further with increasing surfactant concentration (Osipow 1962). However, the ability of surfactant solutions to solubilize water-insoluble materials starts at the cmc and increases with the concentration of micelles (Osipow 1962).

Often, heating an aqueous cleaning solution can improve cleaning efficiency. Generally, the solubility of organic chemicals is greater in heated solutions. Heat also raises the surface temperature, thereby facilitating volatilization of sorbed organic contaminants. In the case of gross contamination with pure product, heat would also reduce the viscosity of contaminants, thereby facilitating physical removal from either the surface or within the pore structure. Keys (1980) compared the effect of spray wash temperature (21°C vs. 49°C, or 70°F vs. 120°F) on the ability of two detergent solutions to remove soil from a surface (Fig. 1).
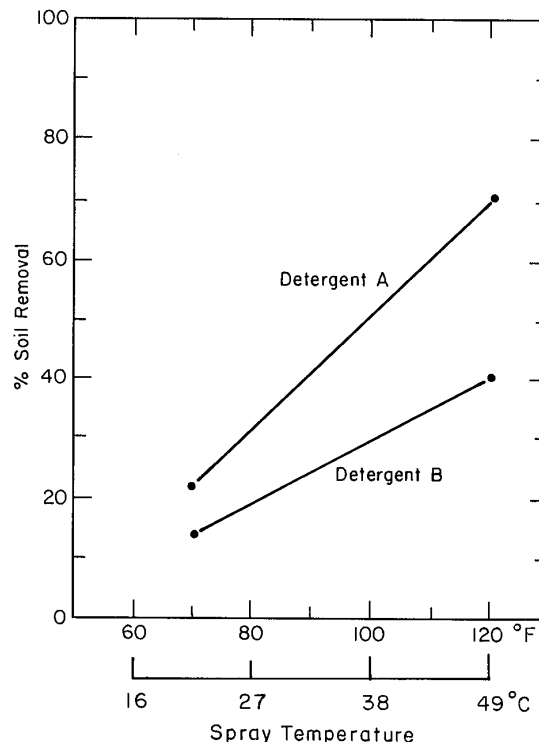


*Figure 1. Effect of spray temperature on soil removal (detergents at 0.5% concentration; 15-sec sprays) (from Keys 1980).*

The hottest water (49°C or 120°F) was more than twice as effective as the coolest water. Studies that have assessed the ability of detergent solutions to remove neat chemical warfare agents from various surfaces (an extreme example of contamination) have also generally found increased removal efficiencies using heated solutions (Mills et al. 1977, Bagley and Weatherhead 1978).

However, according to Osipow (1962), the effect of temperature on surfactant solutions is not straightforward. For ionic surfactants in the absence of other additives, the cmc generally increases with increasing temperature. (Although for some ionic surfactants, there is an optimum temperature that produces a minimum cmc.) This means that, generally, more surfactant will be required in heated cleaning solutions to reach the cmc. However, temperature also affects the solubility of the surfactants in a way that counters this trend. The solubility of these (ionic) surfactants is low at low temperatures and increases slowly and regularly with increasing temperature until, within a narrow temperature range, it increases very rapidly. According to Osipow (1962), this is because ionic surfactants in unassociated

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form have quite limited solubility in water, while the micelles are highly soluble. Thus, ionic surfactants will dissolve more readily and to a greater extent in heated water. The effects of temperature can be quite different for some nonionic surfactants. For some nonionic surfactants, the cmc has been found to decrease with increasing temperature, and others are less soluble in hot water than in cold.

The presence of other substances in the cleaning solution, such as ions (e.g., metals) or organic contaminants, can also affect removal efficiencies. According to Osipow (1962), electrolytes lower the cmc and increase the size and alter the charge of the micelles of ionic surfactants. Thus, electrolytes increase the solubilization of nonpolar materials and may increase or decrease the solubilization of polar materials, depending upon the compound being solubilized (Osipow 1962).

In some studies there is mention of adding organic solvents to cleaning solutions to aid in the dissolution of sorbed organic contaminants. Although not much data on the effectiveness of this approach was found, some examples of organic solvents that have been used for this purpose include ethanol, acetone, and butanol. According to Osipow (1962), added organic compounds increase the micelle volume of ionic surfactants, and the increased volume of this "pseudophase" is expected to have an increased solvent action on other organic materials. (Concentrations of examples given by Osipow were 0.1 to 0.3 moles/1000 g surfactant.) Organic compounds may also affect the cmc of surfactants. Long-chain alcohols and amines will lower the cmc, and short-chain alcohols may increase or decrease the cmc depending upon the temperature (Osipow 1962).

*Pressurized water cleaning*

With steam cleaning, pressure developed in the steam boiler imparts a high velocity to a mixture of water droplets and steam, which is directed from a nozzle onto the target surface. Detergent and heat from the steam weaken the bonds between the dirt and surface while the high velocity of the water droplets has sufficient power to remove the debris from the surface (Summers 1982). Depending upon the contaminant, steam can also facilitate volatilization, thermal decomposition, and hydrolysis. Esposito et al. (1985) felt that long-term exposure to steam should theoretically aid in the removal of subsurface contaminants as well. Problems associated with steam cleaning include

**Table 1. Types of pressurized water systems (from Summers 1982).**

| Type | Pressure | Flow rate (L/min) |
|------|----------|-------------------|
| Low pressure (100 psi) | 689 kPa (10 to 100 gal/min) | 38 to 378 |
| Intermediate pressure (500 to 3,000 psi) | 3445 to 20,700 kPa (5 to 25 gal/min) | 18.8 to 94.5 |
| High-pressure jets (10,000 to 20,000 psi) | 68.9 to 138 MPa (5 to 25 gal/min) | 18.8 to 94.5 |

burns, corrosion of metal surfaces, and liberation of toxic vapors (if sufficient quantities are present).

With high-pressure water washers, high-pressure pumps produce solid water streams rather than the small droplets produced by steam cleaning. The advantages over steam cleaning are that there is increased force available and that the energy requirements are lower because water does not have to be converted to steam. Summers (1982) placed pressurized-water washing systems into three categories: 1) those with relatively low pressure and high flow rates, 2) those with intermediate pressure and flow rates, and 3) those with relatively high pressure jets and intermediate flow rates (see Table 1 for specific pressures and flow rates).

Summers (1982) found that adding long-chain polymers (such as Polyox [Union Carbide], Nalco [Nalco Corp.], and Superwater [Berkley Chemical Corp.]) to the wash water improved performance. Although the function of these polymers is not fully understood, Summers believed that they reduced the flow resistance of the liquids and maintained the integrity of the jet stream for a longer distance after it left the nozzle. The efficiency of high-pressure water jets can also be enhanced with other additives, such as abrasive or nonabrasive particulates (abrasive cleaning), surfactants, or other additives that affect the physics of interaction between the liquid jet spray and contaminant residue (Cante et al. 1980).

**Cleaning or rinsing with organic solvents**

Typically, organic solvents are used where 1) water is undesirable (e.g., for electrical and electronic devices or where humidity might corrode neighboring equipment), 2) contaminants consist of grease, tar, oil, and rosins, 3) fast evaporation is

**Table 2. The most commonly used cold solvent cleaners (from Poliakoff 1973).**

| | |
|---|---|
| 1,2-dichloroethane | methylchloroform |
| acetone | methylene chloride |
| benzene | mineral spirits |
| carbon tetrachloride | perchloroethylene |
| chloroform | toluene |
| ethanol | trichloroethylene |
| isopropanol | trichlorotrifluoroethane |
| methanol | turpentine |
| methyl ethyl ketone | |

needed, or 4) an ultra-clean product is needed for manufacturing purposes (adapted from Poliakoff 1973). Organic solvents work primarily by dissolving the contaminants. Generally like dissolves like. That is, nonpolar solvents dissolve nonpolar organic contaminants, and polar solvents dissolve polar organic contaminants. Table 2 lists the most commonly used solvent cleaners (a large majority of these solvents would not be suitable for cleaning sampling devices because they are target analytes). Thus, solvent cleaners will soften, dissolve, and remove contaminants composed of oils, tars, asphaltics, and other substances that are not readily soluble in water. Materials like salt, soil, and sugars are not readily dissolved by solvent cleaners (Poliakoff 1973).

Solvent cleaning is typically classified as cold cleaning or vapor cleaning. Cold cleaning is done by applying the unheated organic solvent in several ways: 1) wiping the surfaces with a cloth or scrubbing with a brush, 2) immersing the object in some type of tank, 3) spraying the surface with atomized droplets of solvent or flushing it with a solid stream of solvent, and 4) cleaning equipment in place by circulation cleaning (Poliakoff 1973). The first method utilizes agitation and the action of the solvent. For immersion cleaning to be effective, either the piece being cleaned or the solvent should be agitated to ensure rapid cleaning (Poliakoff 1973). However, this process typically leaves some residual contaminated film on the surface because the solvent becomes contaminated in the cleaning process. Using a series of tanks containing cleaner and cleaner solvent can reduce this problem. For spray cleaning, selecting the correct pattern of spray is essential for efficient cleaning (Poliakoff 1973). The first three methods are typically used for smaller items, and the last method is typically used for larger equipment that cannot be easily moved. Poliakoff (1973) also cautions that before using a system that has

been cleaned by circulation cleaning that all the solvent be purged from the system.

If mechanical force, such as high-pressure spraying, hand wiping or brushing, or ultrasonic energy is applied, inorganic contaminants can be scrubbed from the surface by the applied energy (Poliakoff 1973). In the absence of these mechanical aids, solvent cleaners are not effective in removing inorganic and most water-soluble contaminants (Poliakoff 1973). Solvent soaking can be used to remove contaminants that have penetrated a permeable material but this can be a time-consuming process, and the solvent may carry contaminants deeper into the matrix before outward movement occurs (Esposito et al. 1985).

One problem associated with using cold cleaning is that workers will be exposed to vapors of the organic solvents, and many of these solvents are toxic. In addition, the solvents may be flammable. A person working with these chemicals should wear the recommended protective equipment and should be aware that some organic solvents can penetrate some types of protective clothing. Another problem with using organic solvents is that many can degrade the surfaces being cleaned. Many polymers (thermoplastics, thermosets, elastomers, and rubbers) are degraded by organic solvents, especially the chlorinated hydrocarbons. Alcohols are generally gentler on most polymeric surfaces, and most organic solvents do not react chemically with metal surfaces. Disposing of organic solvents is an additional problem unless the solvents can be sent for recycling.

Vapor cleaning is carried out in a chamber where the solvent is boiled and the vapor is recondensed by means of cooling coils. The cold article is placed in the vapor, solvent condenses on it and dissolves soluble contaminants that drain from the surface into the heated solvent where solvent is again vaporized, leaving the involatile contaminants in the heated solvent sump. The process continues until the article has attained the vapor temperature (Wadsworth 1975). Providing the volume of solvent condensed is sufficient, there is no possibility of a residual film (Wadsworth 1975). This is why this method is preferred for items that need to be critically cleaned or ultra-clean.

A number of other novel solvent cleaning concepts exist (e.g., using Freon 113 and supercritical fluids), but these methods would be expensive and do not appear to be either practical or necessary for most groundwater sampling devices.

**Table 3. Decontamination solutions for drilling equipment (from Moberly 1985).**

| Chemical | Solution | Uses/remarks |
|---|---|---|
| Sodium carbonate | 4 lb/10 gal water | Neutralize organic acids, heavy metals, and metal processing wastes |
| Sodium bicarbonate | 4 lb/10 gal water | Neutralize either base or acid (baking soda) contaminants |
| Trisodium phosphate (TSP, Oakite) | 2 lb/10 gal water<br>4 lb/10 gal water | Similar to sodium carbonate.<br>Good for solvents and organic compounds (such as toluene, chloroform, TCE, PCBs) |
| Calcium hypochlorite (HTH) | 8 lb/10 gal water | Disinfectant, bleaching, and oxidizing agent. Used for pesticides, fungicides, chlorinated phenols, dioxins, cyanides, ammonia, and other nonacidic inorganic wastes. |
| Hydrochloric acid | 1 pt/10 gal water | Used for inorganic bases, alkali and caustic wastes |
| Citric, tartaric, and oxalic acids (or their respective salts) | 4 lb/10 gal water | Used to clean heavy metal contamination. |
| Organic solvents (e.g., acetone, methanol, methylene chloride) | Concentrated | Used to clean equipment contaminated with organics or surface oils. |

## Chemical neutralization methods

A number of chemicals can be used to neutralize various organic contaminants. Although this approach is not commonly used to decontaminate sampling devices used for monitoring groundwater, the military has found this approach necessary when decontaminating surfaces contaminated with neat chemical warfare agents. Moberly (1985) outlined a number of chemicals that could be used for decontaminating drilling equipment (Table 3). Bleaching agents include calcium hypochlorite (HTH), Super Tropical Bleach (STB), and household bleach. Bleaches can degrade and detoxify many of the more toxic contaminants, such as organo-phosphorus pesticides (Esposito et al. 1985). Two problems with this type of approach are that many of these decontaminants are hazardous to work with and they may degrade the surface being decontaminated. For example, bleaches can corrode metal equipment. Thus, this approach is really only applicable for extreme cases where one encounters high levels of very toxic contaminants or where other cleaning methods have proven ineffective in reducing contaminant levels.

## CURRENT PROTOCOLS FOR DECONTAMINATING GROUNDWATER SAMPLING DEVICES

Commonly, guidelines for decontaminating sampling devices (e.g., U.S. EPA 1992) suggest that equipment be decontaminated by first washing with a nonphosphate detergent, then rinsing with tap water, rinsing with (high-quality) acids and solvents, rinsing with some type of high-quality water (e.g., distilled, deionized, organic-free reagent water), and finally air drying. Washing with detergent and water removes gross contamination and particles, an acid rinse desorbs inorganic contaminants, a rinse with organic solvents removes less polar organic contaminants, and the final high-quality water rinse removes the acid solutions and/or organic solvents.

Almost all methods recommend either using a nonphosphate (e.g., ASTM 1990, U.S. EPA 1992) or a laboratory detergent (e.g., U.S. EPA Region IV 1991), or both. Although the surfactants used in detergents can be cationic, anionic, amphoteric, or nonionic, there has been little discussion of

**Table 4. Properties of four detergent solutions.[1]**

| Detergent | % phosphate (as P) | Type of detergent | pH of 1% sol. | Recommended (%) conc's, for different water temps | | |
|---|---|---|---|---|---|---|
| | | | | Cold | Warm | Hot |
| Alconox | 7.3 | anionic | 9.5 | 1 | 1* | 1[†] |
| Liquinox | 0 | anionic | 8.5 | 1 | 1* | 1[†] |
| Detergent 8 | 0 | nonionic | 11.0 | 3–5 | 2–3* | 2–3*[†] |
| Citranox | 0 | ** | 2.52 | 2 | 2[†] | 2** |

[1] Information provided by Alconox, Inc.
* Recommended rinse temperature
[†] For hard-to-remove contamination, use higher concentrations
** Citranox is a blend of organic acids, anionic and nonionic surfactants, and alkolamines.

which type of nonphosphate or laboratory detergent to select. Presumably it was thought that if the detergent removes similar contaminants from similar types of surfaces in the laboratory, it ought to work in the field. ASTM Method D-5088-90 (ASTM 1990) recommends using a nonphosphate detergent and refers to Alconox and Liquinox by name. Several other methods also refer to Alconox. However, according to the manufacturer, Alconox is not phosphate-free. Several other detergents (Liquinox, Citranox, and Detergent 8) made by the same manufacturer (Alconox, Inc.) are phosphate-free. Table 4 summarizes the characteristics of these detergents. All four of them can be used for soaking, brush cleaning, ultrasonic cleaning, and flow-through cleaning in place (as would be used for a pump), but only Detergent 8 is recommended for spray cleaning. The manufacturer recommends two phosphate-free detergents, Liquinox and Detergent 8, for cleaning equipment that will be used for environmental sampling and testing.

Typically, the acid rinse is dilute (0.1 N) hydrochloric or nitric acid. Most protocols do not recommend this step unless inorganic species are going to be analyzed. Recommended organic solvents vary with the particular protocol but typically include acetone, hexane, or methanol. Obviously, any organic solvent that is used as a rinsing agent should not be one of the target analytes or interfere with chemical analyses. In addition, the waste decontamination fluids need to be disposed of properly and should be containerized, at least until it can be determined if the waste is hazardous. Clearly, it is advantageous to find the most effective decontamination method that generates the least amount of hazardous waste.

**Variations in methodology**

Mickam et al. (1989) compared the decontamination procedures of several state and federal regulatory agencies and found that these procedures were highly variable. Following is a discussion of some (but not all) of the variations that have been found.

While most methods did not stipulate how aqueous cleaning was to be conducted (i.e., soaking, scrubbing with a brush, or using a high-pressure washer), a few methods [ASTM 1990, U.S. EPA Region IV 1991, and according to Mickam et al. (1989) older procedures for Alabama and Pennsylvania] did recommend using a brush during the cleaning process. In the past, a few state agencies have also recommended using either steam cleaning [California, New Jersey, and New York according to Mickam et al. (1989)], or a power washer [Maryland and New Jersey according to Mickam et al. (1989)] either to remove gross contamination or for equipment used for collecting samples to be analyzed for organic contaminants (California). Region IV of the U.S. EPA also recommended steam cleaning when necessary (U.S. EPA Region IV 1991). In addition, several research studies (Bryden et al. 1986, Matteoli and Noonan 1987, Fink and Boyajian 1989) have used steam cleaning to clean sampling equipment, and a paper by Brice and Kelley (1991) recommended using a high-pressure sprayer.

Brice and Kelley (1991) give one of the most descriptive and extensive protocols for aqueous cleaning of sampling devices. They recommended the following cleaning procedure for lightly contaminated equipment: 1) remove particles using a low-volume, high-pressure sprayer, 2) scrub in a phosphate-free detergent solution and rinse

**Table 5. Recommended solvent rinses for soluble contaminants (from U.S. EPA 1991).**

| Solvent | Soluble contaminants |
|---|---|
| Water | short-chain hydrocarbons<br>inorganic compounds<br>salts<br>some organic acids<br>some polar organic compounds |
| Dilute acids | basic (caustic) compounds<br>amines<br>hydrazines |
| Dilute bases (detergent, soap) | metals<br>acidic compounds<br>phenols<br>thiols<br>some nitro and sulfonic compounds |
| Organic solvents, e.g., alcohols, ethers, ketones, aromatics, straight-chain alkanes (e.g., hexane), common petroleum products (e.g., fuel oil, kerosene) | nonpolar organic compounds |

thoroughly with potable water, 3) rinse with deionized or distilled water, and 4) air dry. For grossly contaminated equipment they recommended the following protocol: 1) wash with detergent solutions and rinse with potable water, 2) rinse with an organic solvent (preferably isopropanol), 3) wash with a detergent solution a second time and rinse with potable water, 4) rinse with deionized or distilled water, and 5) air dry. They cautioned that this equipment should not be used to sample water suspected of being contaminated at low or trace levels. However, they did not supply any data to back up either of these recommended methods.

The U.S. EPA (1991) gives different recommended solvent rinses for various contaminants (Table 5). However, the list of recommended organic solvents for removing nonpolar organic contaminants is general, and it is doubtful whether a number of the solvents they recommend should be used. For example, fuel oil or kerosene can both be target analytes.

When sampling for organics, draft guidance by the U.S. EPA for RCRA sites (U.S. EPA 1992) recommends two solvent rinses: one with hexane or methanol, followed by a rinse with acetone, followed by a rinse with organic-free reagent water. When sampling for metals, this document also recommended using a dilute hydrochloric acid rinse

instead of a dilute nitric acid rinse when cleaning stainless steel. They stated that this was because nitric acid may oxidize steel. However, any acid solution can corrode a stainless steel surface (Driscoll 1986). For glass and polymeric surfaces, the purpose of an acid rinse is to remove sorbed metal ions from the surface. This procedure would be of questionable effectiveness for any metal surface.

Region IV of the U.S. EPA (U.S. EPA Region IV 1991) recommended different protocols for cleaning equipment used to collect samples for trace organic compounds and metals, depending upon the material being cleaned. For fluoropolymers (Teflon) and glass they recommended an acid rinse, but for stainless steel and other metal sampling devices they did not. They did not explain why these methods differed. They also gave different protocols for precleaning various types of sampling tubings. They recommended that stainless steel tubing receive a detergent wash and rinse with high-quality water and a rinse with (an unspecified) organic solvent. Silastic rubber tubing would receive a similar wash but would not be rinsed with organic solvent. In this case, the reason is probably because the rubber tubing would be degraded by most organic solvents. The glass and Teflon tubings receive only an organic solvent rinse, followed by air drying for the glass

tubing and oven drying for the Teflon. In this instance, it is not clear why they eliminated the detergent wash.

In fact, very little scientific evidence could be found to justify any of the recommended protocols. Mickam et al. (1989) concluded that the base of research information is insufficient to permit an adequate assessment of these methods. Apparently, decontamination methods are either based on unpublished observations or best guesses.

**Special instructions
for cleaning pumps**

Cleaning a sampling pump is much more involved than cleaning a bailer. With a pump, there are more surfaces and crevices and often there are several different types of materials. Only three methods that dealt specifically with cleaning pumps were found in the literature. These methods recommended either circulating or recirculating a series of cleaning fluids through the pump system. Classen (1982) recommended pumping an acid-wash solution (100–200 L) through the system and rinsing with deionized water in a similar fashion. However, he did not supply any data to prove the effectiveness of this method. Keely and Boateng (1987) recommended recirculating three cleaning solutions: 1) detergent in water (the outside surfaces would be scrubbed), 2) 10% (vol/vol) acetone in clean water (again the outside would be scrubbed), and 3) rinse with distilled water, followed by air drying. For high concentrations of nonvolatile organics and pesticides, they recommended a second distilled water rinse. They claimed that this method was effective for decontaminating a positive-displacement (Johnson–Keck) submersible pump that had been used to sample VOCs, but they did not offer any proof of this. They also claimed that this series of cleaning solutions was effective for cleaning a stainless steel bailer exposed to similar contaminants.

ASTM Method D 5088-90 (ASTM 1990) for decontaminating sampling devices (used at nonradioactive sites) recommends circulating 1) a detergent solution, 2) a rinse of known quality water, 3) a rinse with 10% nitric acid or hydrochloric acid (this step is deleted if samples are not analyzed for inorganic contaminants), 4) a rinse with known-quality water, 5) a rinse with an organic desorbing agent (isopropanol, acetone, or methanol), 6) a rinse with deionized water, and following all of this with air drying. Again, this method did not provide any scientific studies that justify using this procedure.

**EFFECTIVENESS OF VARIOUS
DECONTAMINATION METHODS**

When the literature was reviewed, only five studies (Dunbar et al. 1985, Bryden et al. 1986, Matteoli and Noonan 1987, Fink et al. 1989, Schuh et al. 1993) were found that compared the effectiveness of various decontamination protocols. Of these five studies, only two (Dunbar et al. 1985, Bryden et al. 1986) were in peer-reviewed journals. Bryden et al. (1986) reported having excellent results decontaminating purging and sampling equipment in the field using a small portable steam-cleaning machine followed with a rinse with distilled water or "type II" water (to remove any contaminants coming from the steam cleaner or water supply). They did not elaborate on the types of contaminants they encountered, but reported that the pressure was sufficient to dislodge particulate matter. They also warned that Teflon could warp if steam-cleaned too vigorously.

Dunbar et al. (1985) found that rinsing a stainless steel bailer and nylon rope with clean water was not sufficient to prevent carryover of contaminants (chloroform and benzene) from well to well. They recommended decontaminating the bailer by using steam cleaning, washing with detergent, and rinsing with deionized water, and using new lengths of nylon rope in each well. However, they did not present any data that substantiates this recommendation. Presumably, the bailer would be much easier to decontaminate than the nylon rope.

The next three studies (Matteoli and Noonan 1987, Fink et al. 1989, Schuh et al. 1993) actually compare the effectiveness of various decontamination protocols. Unfortunately none of these studies was peer reviewed. However, because there were no other peer-reviewed studies available and because they do provide us with more complete data, they were included in this discussion.

Matteoli and Noonan (1987) conducted several field tests to study decontamination of sampling tubings. They tested two types of tubing: rubber hose (neoprene lined with ethylene propylene-diene monomer) and fluorinated ethylene propylene (FEP, another type of Teflon). In their first two field tests, they used a stainless steel submersible pump with the rubber hose. They pumped well water contaminated with TCE, at concentrations of approximately 860 to 1100 ppb, for 2 hours. They then steam cleaned the exterior of the pump and hose and pumped clean water

through the system for another 2–3 hours. At the end of this time, they were still able to detect low ppb levels (1.1 to 18) of TCE in water pumped through the system. They concluded that rubber hose could not be decontaminated using this method in an acceptable amount of time.

In all of their subsequent tests they used the same methodology except that they used FEP tubing rather than the rubber hose and the original exposure time was less, 30 minutes. In the third test, they found that TCE concentrations were below the detection limit (0.5 ppb) after pumping clean water for 90 minutes. The initial level of TCE contamination was similar to the previous studies, approximately 1200 ppb. In the next (fourth) trial, they were unable to completely decontaminate the system. They found that, after pumping clean water through the system for 120 minutes, concentrations of Freon 113 (1,1,2-trichloro-2,2,1-trifluoroethane) ranged from 1.7 to 21 ppb; initial concentrations of this analyte were approximately 3900 ppb. In the fifth test, 1,1-dichloroethylene concentrations dropped below the detection limit (0.5 ppb) after pumping clean water for only 7 minutes. However, the initial concentration of this contaminant was much lower, only about 120 ppb. In the sixth test, dimethyl nitrosamine concentrations fell below the detection limit (0.2 ppb) after pumping clean water for only 3 minutes. Again, initial concentrations were low (~30 ppb). The next two tests indicated that metal contaminants are more readily decontaminated. (Selenium concentrations dropped below the detection limit—5 ppb—after pumping clean water for only 3 min; initial concentrations were 610 ppb in the contaminated well. In another test, initial chromium concentrations in the well water were 43,000 ppb. However after pumping with clean water for 20 minutes, concentrations were at the background level.) They concluded that residual TCE concentrations dropped off more rapidly using Teflon than rubber hose. However, given that the initial contact time was 4 times longer for the rubber hose than for the FEP tubing, a valid comparison cannot be made. It is clear from this data that decontaminating either type of tubing by steam cleaning the exterior and pumping clean water through the system can be time-consuming and ineffective for certain organic contaminants, especially if the initial concentrations of the contaminants are in the ppm range and contact times are 30 minutes or more.

Fink et al. (1989) evaluated four methods for decontaminating a sampling pump. This test compared detergent washes with plain water washes. Method 1 involved injecting 30 gal warm water (49°C or 120°F) with detergent (1%), followed by rinsing with 10 gal of ambient-temperature water. Method 2 was similar, except that only half the previous amount of cleaning and rinsing solutions were used (15 gal of detergent wash water and 5 gal of ambient rinse water). Method 3 involved washing with 30 gal warm water (no detergent) followed by a 10-gal rinse with ambient-temperature water. Method 4 was similar except that the amount of water was again half (15 gal warm water, 5 gal ambient-temperature water). The four solutions were pumped through a stainless-steel bladder pump (equipped with a PTFE bladder) and PTFE-lined sample tubing. After removing the pump from the well, the external surfaces of the pump and tubing assembly were steam cleaned before circulating the cleaning fluids through the pump. The pump had been used to sample VOCs; a minimum of five sampling pump/tubing assembly volumes of contaminated water were pumped from the well between decontamination procedures.

In the first study, initial concentrations of TCE in the well water were 300 mg/L. They found that all four methods yielded rinse water with no TCE contamination. In a field test, they found no VOCs in 26 of 28 quality-control samples taken after using method 2 to clean the sampling device. Although they claimed there was no effect of concentration on decontamination efficiency, the data do not support this claim. TCE concentrations of 1.0 µg/L and 1.8 µg/L were found in the two samples that were pumped from the two wells with the highest TCE concentrations (1600 µg/L and 2215 µg/L, respectively). For the rest of the study, initial concentrations of TCE in well water were much lower (all but one sample had concentrations of 300 ppb or less) and initial concentrations of the other VOCs (1,1-dichloroethane, 1,1-dichloroethylene, tetrachloroethylene, and 1,1,1-trichloroethane) were in the low (< 10 µg/L) range.

Schuh et al. (1993) tested the effectiveness of a distilled water rinse for removing seven pesticides [bromoxynil, diclofop-methyl, dimethoate, 2-methyl-4-chlorophenoxyacetic acid (MCPA), methyl parathion, propiconazole, and trifluralin] from a PVC bailer. They found that the effectiveness of this procedure depended on the analyte's solubility in water and its $K_{ow}$ value. Contact time was brief (1 minute); they felt that a 1-min contact time was representative for samples taken in a shallow well or gravelly aquifer. They found

**Table 6. Sorption and desorption of three organic solutes by rigid PVC and PTFE.**

| Material | Analyte | Initial concentration = 2000 µg/L | | | | | |
|---|---|---|---|---|---|---|---|
| | | Contact time = 6 wk (1000 hr) | | Contact time = 24 hr | | Contact time = 1 hr | |
| | | Amt. sorbed* (µg/L) | Amt. desorbed* (µg/L) | Amt. sorbed* (µg/L) | Predicted amt. desorbed (µg/L) | Amt. sorbed* (µg/L) | Predicted amt. desorbed (µg/L) |
| PTFE | CDCE | 410 | 200 | 80 | 39 | ND | — |
| | CLB | 1000 | 280 | 200 | 56 | ND | — |
| | MDCB | 1480 | 355 | 440 | 11 | ND | — |
| Rigid PVC | CDCE | 200 | 80 | 100 | 40 | ND | — |
| | CLB | 280 | 100 | 100 | 36 | ND | — |
| | MDCB | 400 | 200 | 160 | 80 | ND | — |

| Material | Analyte | Initial concentration = 50 µg/L | | | | | |
|---|---|---|---|---|---|---|---|
| | | Contact time = 6 wk (1000 hr) | | Contact time = 24 hr | | Contact time = 1 hr | |
| | | Predicted amt. sorbed* (µg/L) | Predicted amt. desorbed* (µg/L) | Predicted amt. sorbed* (µg/L) | Predicted amt. desorbed* (µg/L) | Predicted amt. sorbed* (µg/L) | Predicted amt. desorbed (µg/L) |
| PTFE | CDCE | 10 | 5 | 2 | 1.0 | ND | — |
| | CLB | 25 | 7 | 5 | 1.4 | ND | — |
| | MDCB | 37 | 9 | 11 | 2.8 | ND | — |
| Rigid PVC | CDCE | 5 | 2 | 2.5 | 1.0 | ND | — |
| | CLB | 7 | 2.5 | 2.5 | 0.9 | ND | — |
| | MDCB | 10 | 5 | 4.0 | 2.0 | ND | — |

\* Actual values taken from Parker et al. (1990). Desorption time was 3 days.
CDCE  =  cis-1,2-dichloroethylene
CLB  =  chlorobenzene
MDCB  =  m-dichlorobenzene
ND  =  no loss detected

that most analytes having a solubility of >500 mg/L or $K_{ow}$ <200 (log $K_{ow}$ <2.3) were generally cleaned from a PVC bailer so there was no residual carryover with just one rinse with distilled water. Analytes having a water solubility of >50 mg/L or $K_{ow}$ <2000 (log $K_{ow}$ <3.3) were generally effectively cleaned from a PVC bailer following multiple (>6 rinses) with distilled water. They found that dimethoate was an exception and that 15 rinses were required before it was removed. They concluded that in some cases it may be justifiable and appropriate to rely on a distilled water rinse alone. For this study, it is important to note that there was no significant loss of these analytes due to sorption by the bailer during the 1-min contact time and that contaminant concentrations were generally less than 20 µg/L. The effectiveness of this method might be quite different if a longer contact time (that allowed sorption to occur) were used or if the concentration of the contaminants were higher (at least for the more water-soluble ones).

These studies indicate the importance of the initial concentration of the contaminants, contact time, and the relative sorptiveness of the material being decontaminated. We can use the data from one of our previous studies (Parker et al. 1990) to demonstrate this. Table 6 shows the amount of three organic solutes [cis-1,2-dichloroethylene (CDCE), chlorobenzene (CLB), and m-dichlorobenzene (MDCB)] sorbed by two rigid polymers (PVC and PTFE) after 6 weeks contact with the test solution (at ~2 ppm concentrations) and the amount desorbed after the test pieces were rinsed with fresh well water and placed in clean vials with fresh well water for three days. This table also shows the amount that was sorbed after 1 hour and 24 hours contact with the test solution. If we use this data and assume that the relative rate of desorption remains the same regardless of the amount sorbed, we can estimate the amount of analyte that might be desorbed from the samples that had contact times of 24 hours and 1 hour. (Twenty-four hours was selected to simu-

late the time a pump might remain in a well. Ideally, 10 minutes would best simulate the contact time of a sample with a bailer. However, since the shortest time interval for which there was data was 1 hour, we used these data.) These data were then used to estimate the amount desorbed if the concentration of the analytes in the test solution had been much lower, e.g., 50 μg/L. It is clear from this table that potential problems with cross contamination due to desorption of analytes is greater for the more sorptive material, PTFE, and decreases substantially for relatively short contact times (24 hr or less).

These values are only estimated, and there are a number of factors that could affect the ability of a material to desorb contaminants. For example, the potential for cross contamination would be less if the time for desorption were more realistic of a sampling event and less than the three days used in this study. On the other hand, more hydrophobic contaminants would be expected to be more readily sorbed; the most hydrophobic contaminants used in our study (Parker et al. 1990) were p- and m-dichlorobenzene, which have log $K_{ow}$ values of 3.39 and 3.38, respectively (Hansch and Leo 1979). For flexible tubings, sorption is considerably more extensive. For example, Gillham and O'Hannesin (1990) found that after only 10 minutes contact time, flexible PVC sorbed 50% of low (~1) ppm levels of m- and p-xylene, and rigid PVC sorbed little or nothing (losses were 2% or less). Thus, for a pump that remains in the well for several hours, a large amount of some analytes could be sorbed by the tubing. These contaminants would be available for desorption and could cause problems with cross contamination. A recent study by our laboratory shows that all of the 20 types of tubing we tested (including several fluoropolymers) were highly sorptive of some organics (Parker and Ranney, in press).

Given the previous example (Table 6), it is understandable how rigid materials subjected to low ppb concentrations of relatively water-soluble contaminants for short periods of time may be easily decontaminated with a single aqueous rinse. It is also understandable how higher concentrations of these contaminants or similar concentrations of more hydrophobic contaminants may require several rinses or more stringent cleaning (e.g., detergent cleaning). For flexible materials, such as pump tubing where contact times would be much longer than with a bailer, it is clear that either more extensive cleaning will be necessary or that new or dedicated tubing may be required

for each well to reduce the possibility of cross contamination, especially for the more hydrophobic contaminants and when concentrations exceed more than a few ppb.

## LOW-TEMPERATURE CONSIDERATIONS

While the primary focus of this literature review has been on determining the efficacy of various decontamination methods, the effect of cold temperatures on this process should also be mentioned. As might be expected, subfreezing temperatures limit the usefulness of aqueous and solvent-based decontamination methods. Richter and Collentine (1983) reported that decontamination of field equipment in remote areas of Wyoming was frustrating and that subfreezing temperatures of –40°F rendered conventional methods with soap and water impossible. They recommended taking additional clean equipment in the field so that decontamination can be conducted later in a specially constructed (i.e., heated) wash area.

## CONCLUSIONS

From these studies the following conclusions can be made:

1) Although there are a number of published methods for decontaminating sampling devices, there is a lot of disparity between the methods.
2) There has been very little systematic study of the effectiveness of various decontamination protocols and apparently little documented basis for the existing guidance.
3) The type of contaminants, level of contamination, and materials being decontaminated clearly affect desorption and decontamination effectiveness.
4) To clean sampling pumps with flexible tubings (even Teflon tubings), neither a clean water wash nor a wash with warm detergent followed with a clean water rinse is generally effective if the pump was exposed to ppm levels of VOCs. Steam cleaning with a detergent solution may be more effective, although this remains untested.
5) Cleaning bailers that have very short contact times with the samples appears to be much less problematic.

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6) Using the aqueous solubility or $K_{ow}$ values of the contaminants or multiple linear solvation energy relationships may be useful in predicting the ability to decontaminate a sampling device.

Clearly, further systematic study that evaluates various decontamination methods is needed. These studies need to consider the type of material(s) being decontaminated, a range in hydrophobicity of the contaminants, concentrations of contaminants, and contact time. The kinetics of desorption also need to be addressed.

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**13. ABSTRACT** (*Maximum 200 words*)

Current protocols for decontaminating devices used to sample groundwater for organic contaminants are reviewed. Most of the methods given by regulatory agencies provide little scientific evidence that justify the recommended protocols. In addition, only a few studies that actually compared various decontamination protocols could be found in the open literature, and those studies were limited in their scope. Various approaches for decontamination and criteria that are important in determining how effectively a surface could be decontaminated are discussed.

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Decontamination
Organic contaminants

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