OhioEPA

State of Ohio Environmental Protection Agency

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October 26, 2007

Mr. Robert Schoepke, P.G.
Director - Remediation
Safety-Kleen Systems, Inc.
1502 East Villa Street
Elgin, IL 60120

Re: Safety-Kleen Systems, Inc.
Hebron Recycle Center
TSD/LQG
OHD980587364/01-45-0518
Licking County, CDO
NOV

Dear Mr. Schoepke:

On July 13, 2007, Ohio EPA received a response from Safety-Kleen Systems, Inc. (Safety-Kleen), dated July 12, 2007, regarding Ohio EPA's June 7, 2007, notice of violation (NOV) letter for the Hebron Recycle Center (Facility) in Licking County. The June 7, 2007, letter provided notice of two specific violations of Safety-Kleen's hazardous waste permit and eight general comments regarding permit compliance and recent corrective action activities and information collected at the site. In addition, on July 18, 2007, Ohio EPA received your response to our General Comment 2 contained in the NOV letter regarding vinyl chloride.

Our review of this documentation reveals that Safety-Kleen remains in violation of its hazardous waste permit. The following is our response to your July letters and clarification of Ohio EPA's position on the outstanding ground water issues, including permit compliance and ground water sampling.

Safety-Kleen's July 12, 2007 letter

S-K Response to OEPA NOV Comment (1)

As stated in Safety-Kleen's response to OEPA NOV Comment (1), Safety-Kleen believed that it was performing the sampling for metals in accordance with the intent of the modified permit. The event was performed in accordance with Section 10 of the OEPA-approved revised permit application, which indicated sampling for dissolved metals. However, Permit Condition E.9(a)(vi)(b) states that metals be analyzed in both unfiltered and filtered samples (for those samples with above 5 NTUs).

Ohio EPA's *Technical Guidance Manual for Hydrogeologic Investigations and Ground Water Monitoring, Chapter 10, Ground Water Sampling, February 2006* (TGM) provides guidance on filtration on page 10-35. The TGM does not preclude sites from taking unfiltered samples; it only provides Ohio EPA's general recommendations on whether and how to filter. Safety-Kleen's permit, however, requires analyzing metals in both unfiltered and filtered samples.

U.S. EPA also recommends a dual sampling approach be used for assessing mobility of metals in ground water when high turbidity exists (i.e., collection of both filtered and unfiltered samples). In addition, U.S. EPA recommends filtration of samples should be completed with a filter of a nominal pore size smaller than 0.45 microns. See attached guidance "Superfund Ground Water Issue, Ground Water Sampling for Metals Analyses." Safety-Kleen indicated that a 5-micron filter had been used previously. Please note that the TGM erroneously states on page 10-37 that use of a 5 micron filter is recommended to ensure that the mobile fraction of turbidity is sampled. This is a misprint, as Ohio EPA recommends using a 0.5 micron filter.

Safety-Kleen also stated in its response that the wells were purged and then allowed to stabilize overnight in order to reduce potential turbidity. This is not an appropriate sampling method as discussed with Ohio EPA's Division of Drinking and Ground Waters (DDAGW) per an October 25, 2007, phone conversation. Ohio EPA recommends that Safety-Kleen use a low flow purge and sample method to minimize turbidity. In addition, Safety-Kleen's Part B permit application already allows the use of micropurge methods on page 5-1-7 (Appendix 5-1, Groundwater Monitoring Plan).

Safety-Kleen must comply with the terms and conditions of its permit. Permit condition A.1(a) states that, "The permit application is hereby incorporated into this permit. In the instance of inconsistent language or discrepancies between the above, the language of the more stringent provision shall govern." Thus, Safety-Kleen remains in violation of the following hazardous waste rule and permit condition:

Violation #1

OAC Rule 3745-50-58(A) and Permit Condition E.9(a)(vi)(b), Ground Water Sampling: The permittee must comply with all conditions of the hazardous waste permit. Pursuant to Permit Condition E.9(a)(vi)(b), Safety-Kleen must analyze for the presence of metals in unfiltered samples and should also collect filtered samples from wells in which a turbidity level exceeds five (5) nephelometric turbidity units (NTUs) despite reasonable care to minimize turbidity.

Based on discussions at the June 1, 2007, Ohio EPA/Safety-Kleen meeting, Safety-Kleen did not analyze for the presence of metals in unfiltered samples. Instead, ground water samples were taken, sent to the laboratory, and then filtered before analysis.

☛ To return to compliance, Safety-Kleen must resample all of the wells listed in Permit Condition E.9(a)(vi)(b), analyze for the presence of metals in unfiltered samples, and provide Ohio EPA with the results. Ohio EPA recommends that Safety-Kleen use a low flow purge and sample method to minimize turbidity. Safety-Kleen may resample the wells as part of the next ground water sampling event scheduled for November 2007.

S-K Response to OEPA NOV Comment (2)

As stated in Safety-Kleen's response to OEPA NOV Comment (2), Safety-Kleen does not believe that confirmation sampling for metals is necessary/warranted at this time and refers to the approved Class 2 permit modification request included in Section 10.4.5 of the revised permit application, which states that Safety-Kleen may perform confirmation sampling.

As discussed with Ohio EPA per an October 25, 2007, phone conversation, confirmation sampling is required by the permit independent of the requirement to establish background concentrations, which is in Permit Condition E.9(a)(vii)(b).
Safety-Kleen must comply with the terms and conditions of its permit. Permit condition A.1(a) states that, "The permit application is hereby incorporated into this permit. In the instance of inconsistent language or discrepancies between the above, the language of the more stringent provision shall govern." Thus, Safety-Kleen remains in violation of the following hazardous waste rule and permit condition:

Violation #2

OAC Rule 3745-50-58(A) and Permit Condition E.9(a)(vii)(a), Ground Water Confirmation Sampling: The permittee must comply with all conditions of the hazardous waste permit. Permit Condition E.9(a)(vii)(a) requires Safety-Kleen to conduct confirmation sampling within thirty (30) days of the date the data is reported and validated.

Confirmation samples for metals were not conducted for any of the 13 downgradient monitoring wells subsequent to the initial modified appendix sampling conducted in April 2006, although confirmation sampling was conducted for volatile organic compounds (VOCs). In addition, confirmation sampling was conducted in the background wells, but not for the appropriate metals samples.

- To return to compliance, Safety-Kleen must resample all of the wells listed in Permit Condition E.9(a)(vi)(b) to analyze for metals. If the sampling results in the detection of any metals, Safety-Kleen must conduct confirmation sampling within 30 days of the date the data is reported and validated and provide the results to Ohio EPA. Safety-Kleen may resample the wells as part of the next ground water sampling event scheduled for November 2007.

In General, Comment 1 in Ohio EPA's July 7, 2007 NOV letter, we asked for more information to determine whether Safety-Kleen is in compliance with OAC Rule 3745-50-58(A) and Permit Condition G.7(c). To date, we have not received documentation to justify why the upper prediction limit used was ten times higher than the listed concentration limit. Thus, Safety-Kleen is in violation of the following hazardous waste rule and permit condition:

Violation #3

OAC Rule 3745-50-58(A) and Permit Condition G.7(c), Statistical Procedures: The permittee must comply with all conditions of the permit. Permit Condition G.7(c) requires Safety-Kleen's statistical procedures to be protective of human health and the environment, provide reasonable confidence that the migration of hazardous constituents from a regulated unit into and through the aquifer will be indicated, and determine whether such leakage of hazardous constituents into the ground water exceeds specified concentration limits.

For vinyl chloride, the statistical evaluation needs to be capable of evaluating chemicals of concern (COC) detections to the concentration limit of 2 ug/L as listed in the table in Permit Condition G.2(a). However, Appendix C-1, Table 33B of the Supplementary Annual Report for 2006 indicates that the upper prediction limit for vinyl chloride was 20 ug/L in well H-15S.

- To return to compliance, Safety-Kleen must resample the well for vinyl chloride and use statistical procedures capable of evaluating detections to the concentration limit of 2 ug/L as listed in the table in Permit Condition G.2(a). Safety-Kleen may resample the well as part of the next ground water sampling event scheduled for November 2007 and submit the results to Ohio EPA in the Supplementary Annual Report for 2007.

Safety-Kleen's July 18, 2007 letter

On July 18, 2007, we received your response to OEPA General Comment (2) regarding the significant increase in vinyl chloride concentrations observed in the area of well H-15S. As stated in the letter, the concentration was inadvertently misreported at 86J ug/L. The correct concentration should have been 0.86 ug/L. We will place a copy of the corrected laboratory data report in our files.

Should you have any questions, please feel free to call me at (614) 728-3887. You will find copies of the rules and other information on the division's web page at: http://www.epa.state.oh.us/dhwm.

Sincerely,

Melissa Musko
Environmental Specialist
Division of Hazardous Waste Management
Central District Office

c: Stephen Lear, Safety-Kleen Systems, Inc.
Tammy McConnell, DHWM/CO
Randy Sheldon, DHWM/CDO
Jason Reed, DDAGW/CDO
CDO File

MM/nsm SK.GW.response to July 2007letter

NOTICE:
Ohio EPA's failure to list specific deficiencies or violations in this letter does not relieve your company from having to comply with all applicable regulations.

United States Environmental Protection Agency | Office of Research and Development | Office of Solid Waste and Emergency Response | EPA/540/4-89/001 March 1989

EPA Superfund Ground Water Issue

Ground Water Sampling for Metals Analyses

Robert W. Puls and Michael J. Barcelona

The Regional Superfund Ground Water Forum is a group of ground-water scientists, representing EPA's Regional Superfund Offices, organized to exchange up-to-date information related to ground-water remediation at Superfund sites.

Filtration of ground-water samples for metals analysis is an issue identified by the Forum as a concern of Superfund decision-makers. Inconsistency in EPA Superfund cleanup practices occurs where one EPA Region implements a remedial action based on unfiltered ground-water samples, while another Region may consider a similar site to be clean based on filtered ground-water samples. RSKERL-Ada and EMSL-Las Vegas have convened a technical committee of experts in the areas of ground-water geochemistry, inorganic chemistry, colloidal transport and ground-water sampling technology to examine this issue and provide technical guidance based on current scientific information.

Members of the committee were Robert W. Puls, Bert E. Bledsoe and Don A. Clark of RSKERL; Michael J. Barcelona, Illinois State Water Survey; Phillip M. Gschwend, Massachusetts Institute of Technology; Terry F. Rees, USGS-Denver; John W. Hess, Desert Research Institute (EMSL-LV); and Nicholous T. Loux, ERL-Athens.

This document was written by Robert W. Puls and Michael J. Barcelona and edited by all members of the committee.

For further information contact Robert Puls, RSKERL-Ada, FTS 743-2262; Bert Bledsoe, RSKERL-Ada, FTS 743-2324; Jane Denne, EMSL-LV, FTS 545-2655.

The findings and recommendations of the committee were that use of a 0.45 micron* filter was not useful, appropriate or reproducible in providing information on metals mobility in ground-water systems, nor was it appropriate for determination of truly "dissolved" constituents in ground water. A dual sampling approach was recommended, with collection of both filtered and unfiltered samples. If the purpose of the sampling is to determine possible mobile contaminant species, the unfiltered samples should be given priority. This means that added emphasis is placed on appropriate well construction methods, materials and ground-water sampling procedures. For accurate estimations of truly "dissolved" species concentrations, filtration with a nominal pore size smaller than 0.45 microns was recommended. It was further concluded that filtration could not compensate for inadequate construction or sampling procedures.

Background/Support Information

Filtration of ground-water samples for metal analyses will not provide accurate information concerning the mobility of metal contaminants. This is because some mobile species are likely to be removed by filtration before chemical analysis. Metal contaminants may move through fractured and porous media not only as dissolved species, but also as precipitated phases, polymeric species or adsorbed to inorganic or organic particles of colloidal dimensions. Colloids are generally considered as particles with diameters less than 10 microns (Stumm and Morgan, 1981). Numerous investigators have suggested the facilitated transport of contaminants in association with mobile colloidal particles. Kim et al. (1984) suggested that sorption to ground-water colloidal material caused the mobilization of some radionuclides in Gorleben ground waters. Saltelli et al. (1984) studied americium percolation in glauconitic sand columns and attributed the unretained fractions to migrating colloidal species.

* Micron = μm = 10^{-6} meter

Superfund Technology Support Centers for Ground Water

Robert S. Kerr Environmental Research Laboratory
Ada, OK

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Las Vegas, NV

These colloids were either homogeneous hydrous precipitates, or were formed from the adsorption of the radionuclide onto colloidal size mineral particles. Colloidal particles generated in batch experiments by Sheppard et al. (1979) were shown to adsorb significant quantities of radionuclides. Further work by Sheppard et al. (1980) concluded that the transport of radionuclides by colloidal clay particles must be considered in any contaminant transport model. Champlin and Eichholz (1968) showed that the movement of radioactive sodium and ruthenium in sand beds was associated with particulate matter of micron dimensions. Gschwend and Reynolds (1987) demonstrated that submicron ferrous phosphate colloids were suspended and presumably mobile in a sand and gravel aquifer.

Studies by Yao et al. (1971) and O'Melia (1980) indicate that colloidal particles in the range 0.1 to 1.0 micron may be most mobile in a sandy, porous medium. Kovenya et al. (1972) concluded that particles in the range 0.1 to 0.5 mm were most mobile in soil column studies. As much as 200 ppb copper, lead and cadmium was found associated with colloidal material in size range 0.015-0.450 mm by Tillekeratne et al. (1986). Rapid transport of plutonium (Pu) in core column studies by Champ et al. (1982) was attributed to colloidal transport, with 48% of the Pu associated with colloids in the size range 0.003-0.050 mm and 23% in the range 0.050-0.450 mm. Reynolds (1985) using carboxylated polystyrene beads ranging from 0.10 to 0.91 mm in size, recovered 45% of the 0.91 mm size beads, and greater than 70% of 0.10 and 0.28 mm size beads in laboratory sand column effluents.

Lake and estuarine studies by Baker et al. (1986) and Means and Wijayaratne (1982) demonstrated the importance of natural colloidal material in the transport of hydrophobic contaminants. Carter and Suffet (1982) found that a significant fraction of "dissolved"DDT in surface waters was bound to colloidal humic material. Takayanagi and Wong (1984) found over 70% of the total inorganic colloidal particles.

Analytical methods used to determine "dissolved" metal concentrations have historically used 0.45 micron filters to separate dissolved and particulate phases. If the purpose of such determinations is an evaluation of "mobile" species in solution, significant underestimations of mobility may result, due to colloidal associations. On the other hand, if the purpose of such filtration is to determine truly dissolved aqueous species, the passage of colloidal material less than 0.45 microns in size may result in the overestimation of dissolved concentrations (Bergseth, 1983; Kim et al. 1984; Wagemann and Brunskill, 1975). Kennedy et al. (1974) found errors of an order of magnitude or more in the determination of dissolved concentrations of aluminum, iron, manganese and titanium using 0.45 micron filtration. Sources of error were attributed to filter passage of fine-grained clay particles. Additionally, filtration of anoxic ground-water samples is very difficult without iron oxidation and colloid formation, causing a removal of previously dissolved species to be filtered. Filter loading and clogging of pores with fine particles may also occur, reducing the nominal size (Danielsson, 1981). Filtration should be viewed as only one approach for determining the "true" solution geochemistry of ground water, and others should be applied whenever possible.

Purpose of Sampling

It is important to identify the purpose of ground-water sampling before decisions regarding filtration, centrifugation or other phase separation techniques are made. Is it to determine the mobility of contaminants or to determine in situ aqueous geochemistry? The following definitions are also useful for consideration of this issue:

(1) Total Contaminant Load Per Unit Volume of Aquifer = Mobile + Immobile Species.

(2) Mobile Species = Dissolved + Suspended Species.

(3) Dissolved = Free Ions + Inorganic Complexes + Low Molecular Weight Organic Complexes.

(4) Suspended = Adsorbed + Precipitated + Polymeric + High Molecular Weight Organic Complexes.

For an assessment of mobility, all mobile species must be considered, including suspended particles acting as adsorbents for contaminants. While not all suspended species may necessarily be sufficiently mobile or toxic to pose a health risk, a conservative approach is proposed at this time until more definitive data are available. Contaminant transport models which account for an additional aqueous mobile colloidal phase have been proposed by Avogadro and DeMarsily (1984) and Enfield and Bengsston (1988).

A principle objective in a sampling effort for testing a geochemical speciation model is to obtain estimates of the free ion activities of the major and trace elements of interest. Since there are relatively few easily performed analytical procedures for making these experimental estimates, an alternative procedure is to test the analytically determined dissolved concentrations with model predictions including both free and complexed species. More and more remedial investigations are utilizing such models to make predictions about contaminant behavior based on dissolved concentrations. It is not the purpose of this report to suggest how to perform these analytical determinations, but as noted above, the use of a 0.45 micron filter as the operational definition of "dissolved" may be inappropriate. Analytical techniques such as ion selective electrodes, ion exchange and polarography may be more accurate. Research utilizing these and other techniques to correlate "dissolved" with filter size is recommended.

If one adopts the conservative approach with no filtration for contaminant mobility estimations, increased importance is placed on proper well construction, and purging and sampling procedures to eliminate or minimize sources of sampling artifacts.

Sources of Sampling Artifacts vs. "real" Ground-water Environment

The disturbance of the subsurface environment as a result of well construction and sampling procedures presents serious obstacles to the interpretation of ground-water quality results. Some degree of disturbance of natural conditions is inevitable. However, the impact of improper well construction and sampling techniques can permanently bias the usefulness and integrity of wells as sampling points. Several aspects of well construction and sampling procedures must be carefully considered to avoid errors associated with the introduction of foreign particles or the alteration of ambient subsurface conditions which may affect natural dissolved or suspended materials.

Well Construction

The design, drilling, and construction of monitoring wells have been identified as particularly important steps in the collection of representative water chemistry and hydrologic data. Several references have emphasized the minimization of both the disturbance and the introduction of foreign materials (USEPA, OSWER-9950.1, 1986; Barcelona, et al., 1983; Barcelona et al., 1985) because of the potential impact on water chemistry. The RCRA Technical Enforcement Guidance Document (USEPA, OSWER-9950.1, 1986) suggests that the well must allow for sufficient ground-water flow for sampling, minimize passage of formation materials into the well, and exhibit sufficient structural integrity to prevent collapse of the intake structure. It should be recognized, however, that the well must first provide a representative hydraulic connection to the geologic formation of interest. Without the assurance of this hydraulic integrity, the water chemistry information cannot be interpreted in relation to the dynamics of the flow system or the transport of chemical constituents.

More specific guidance is therefore necessary to maintain or restore the natural hydraulic conductivity of the formation in the vicinity of the screened portion of monitoring wells through the drilling, construction and development procedures. The literature on water well technology can be most helpful in this regard since minimal disturbances of the subsurface is a common goal in maximizing both the yield of water supply wells and the representativeness of water samples and hydraulic information from monitoring wells (Driscoll, 1986).

To insure the long-term integrity of monitoring wells, particularly with respect to excluding foreign particles and permitting the passage of mobile (i.e., dissolved and suspended) contaminants, specific items which should be observed are:

1) If no alternative to the use of drilling muds or fluids exists, these materials must be removed from the well bore and adjacent formations by careful well development (Driscoll, 1986). This guidance also applies to the removal of the low permeability "skin" which is caused by abrasion, oxidation and invasive muds which may seal the well bore from the screened interval and bias in situ determinations of hydraulic conductivity (Faust and Mercer, 1984; Moench and Hsieh, 1985; Faust and Mercer, 1985). Pumping rates during development should be documented and care should be taken not to exceed these rates during purging or sampling since further development and well damage may aggravate suspended particulate and turbidity problems even in properly designed wells.

2) The emplacement of grouts and seals to isolate the screened interval must be carefully done. The use of tremie pipes and frequent checking of the depth of emplacement of clay or cement grouts during well construction are strongly encouraged.

It is also important to take care to follow manufacturer's guidelines on the hydration of cement or expanding cement as grouts or seals. Excess water addition and grading of cement components or materials due to free fall through standing water can permanently damage the well's integrity (Evans and Ellingson, 1988).

3) Casing and screen materials must be selected to retain their integrity in the subsurface environment (i.e., avoid iron, steel), minimize bias to water samples and insure that screen openings are not reduced by the buildup of corrosion products or by compression (USEPA, OSWER-9950.1, 1986). These effects can be checked by repeat determinations of in situ hydraulic conductivity over the useful life of the well. Redevelopment and replacement of the well should be considered if deterioration or significant changes in hydraulic conductivity are observed. Erratic water level readings and sudden changes in turbidity or purging behavior of monitoring wells prior to sampling are warning signs of possible loss of material integrity.

4) Well design fundamentals with regard to the selection of a filter pack and screen size are among the most important issues in obtaining representative hydraulic and water quality information. The exclusion of fines, clays, and silts can be achieved by selecting the grain-size distribution for the filter pack by multiplying the 50-percent retained size of the finest formation sample by a factor of two (Driscoll, 1986). The filter pack material should be cleaned and washed free of fines to insure that extraneous contaminants or particles are removed. The well screen slot openings should be chosen to retain 90% of the filter pack material after development. In natural packed wells it may be advisable to select a screen slot size which will retain at least 50% of the finest material in the screened interval. Minimizing slot screen width however, often leads to greater time and energy spent in well development. The need to document well development procedures cannot be overemphasized.

Maintenance of the hydraulic performance of monitoring wells and the connection of wells to the zones of greatest hydraulic conductivity, where contaminant transport is most probable, should take equal importance to the collection of representative water quality data.

Purging and Sampling

Water that remains in the well casing between sampling periods is unrepresentative of water in the formation opposite the screened interval. It must be removed by purging or isolated from the collected sample by a packer arrangement prior to the collection of representative water samples. Water level readings must be made carefully to avoid the disturbance of fines or precipitates which may enter or form in the well due to chemical reactions or microbial processes and accumulate on the interior walls of the well casing screen or at the bottom of the well. Similarly, it is important to purge the stagnant water at flow rates below those used in development to avoid further development, well damage or the disturbance of accumulated corrosion or reaction products in the well. The use of certain sampling devices, particularly ballers and air-lift arrangements, should be discouraged in order to avoid the entrainment of suspended materials which are not representative of mobile chemical constituents in the formation of interest.

A note of caution should be voiced to encourage repetitive sampling of monitoring wells prior to judging the representativeness of determinations of hydraulic conductivity, water level readings and water quality data. The effects of the inevitable "trauma" due to drilling, sealing and development of monitoring wells can bias observations of water chemistry until the subsurface is allowed to equilibrate sufficiently (Walker, 1983). Estimates of the time to achieve equilibration vary substantially, particularly when drilling fluids are used in highly permeable formations

(Brobst, 1984; Driscoll, 1986); however, periods of weeks to several months may be necessary before even major ionic constituents of ground water equilibrate to previous levels (Barcelona, et al., 1988).

Recommendations for Sampling

In general, the zone of interest must be isolated, the sample pumped slowly to minimize turbidity and sample collected in such manner as to eliminate O_2 and CO_2 exchange with the atmosphere. No filtration for mobile metals determination is recommended. If the unfiltered values exceed maximum contaminant level concentrations for ground-water quality, additional analyses and re-evaluation of sampling artifacts are required. It should be emphasized that extreme differences between unfiltered and 0.45 mm filtered samples does not preclude the use of unfiltered data for risk assessment decisions. Significant particulate mobility may be occurring at such a site, and additional analyses with other larger filters (e.g. >0.45 mm) may be most appropriate given the current size estimates for upper limits for mobile particles.

Isolation of Sampling Zone

Isolation of the sampling zone is necessary to minimize the purge volume as well as to minimize air contact. This is especially important since Eh/pH conditions of the formation waters are notoriously sensitive to dissolved gases content. Inflatable packers can be used to achieve isolation of the sampling zone.

Pumping for Sample Collection

It is recommended that a positive displacement pump can be used. Other types of sample collection (e.g., bailing) may cause displacement of non-mobile particles or significantly alter ground water chemistry leading to colloid formation (e.g., vacuum pumps). Surging must be avoided, and a flow rate as close to the actual ground-water flow rate should be employed. Acknowledging that this may be impossible or impractical in some instances, a pumping flow rate based on the linear ground-water flow rate and open screen area is proposed, where

pumping flow rate ~linear GW flow rate x 2 x screen ht. x well radius x 10

While an initial approximation, flow rates around 100 ml/min have been used to successfully sample ground-waters in a quiescent mode.

Additional research is needed in this area, particularly with respect to the appropriateness of this generic equation. An inexpensive flow-through type cell set-up utilizing this approach was described by Garske and Schock (1986).

Assessment of Water Constituents While Sampling

Monitoring of the pumped ground water for dissolved oxygen, temperature, conductivity and pH aids in the interpretation or establishment of ground-water background quality. Gschwend and co-workers (personal communication) have observed that turbidity diminished dramatically after prolonged pumping, changing similarly, although possibly more slowly, than other water quality parameters (e.g., O_2, conductivity). An initial estimate proposed for time of pumping necessary to collect water from a formation is around two times the time required to get plateau values for the above parameters.

No Filtration for Mobile Fraction Determination

Those samples intended to indicate the mobile substance load should not be filtered. Steps to preserve their integrity, such as acidification, should be performed as soon as possible.

Filtration for Specific Geochemical Information

Any filtration for estimates of dissolved subsurface species loads should be performed in the field with no air contact and immediate preservation and storage. In-line pressure filtration is best with as small a filter pore size as practically possible (e.g., 0.05, 0.10 micron). Using a smaller pore size filter will require longer sample collection time, increasing the need for air exclusion from the sample (Laxen and Chandler, 1982; Holm et al., 1988). Polycarbonate membrane-type filters with uniform and sharp size cutoffs are recommended to minimize particle loading on the filter. Although membrane filters are more prone to clogging than fiber-type filters, the uniform pore size, ease of cleaning, and minimization of adsorptive losses from the sample tend to improve the precision and accuracy in the analytical data. The filter holder should be of material compatible with the metals of interest. Holders made of steel are subject to corrosion and may introduce non-formation metals to samples. Large diameter filter holders (e.g., > 47 mm) are recommended to reduce clogging and pore size reduction and for ease of filter pad replacement. The use of disposable in-line filters are suggested for convenience if of sufficient quality. Prewashing of filters should be routinely performed. Work by Jay (1985) shows that virtually all filters require prewashing to avoid sample contamination.

Quality assurance and quality control becomes increasingly important when adopting the above recommendations. The use of field blanks and standards for field sampling is essential. Field blanks and standards enable quantitative correction for bias due to collection, storage and transport. Analysis of the filters themselves and their particulate load is suggested as a check on mass balance and filtration effects on solid/solution separation efficiency.

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