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**SAMPLING AND ANALYSIS OF  
GROUND WATER QUALITY AT  
HAZARDOUS WASTE SITES IN EASTERN CANADA**

by

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## ABSTRACT

Ground-water quality data from five hazardous waste sites in Eastern Canada are reviewed and the contaminants of concern are identified. At three of the sites, contamination is due to the presence of dense, non-aqueous phase liquids (DNAPLs); DNAPLs are suspected at the other two. These have partially dissolved to create plumes of chemicals comprising the contaminants of concern, e.g., halogenated aliphatics, various aromatics and PCBs. Methods of field sampling, preservation and analysis for such polluted ground waters are described. The use of a coupled gas chromatograph-mass spectrometer (GC-MS) for quantitative organic analysis of ground-water pollutants is presented and the particular problem of the analysis of heavily polluted samples is discussed. The principal elements of NWRI's quality assurance/quality control program for heavily polluted ground waters is explained, with an emphasis on obtaining data of high precision and accuracy in spite of matrices of great complexity.

## RÉSUMÉ

Ce rapport examine les données sur la qualité des eaux souterraines de 5 sites d'enfouissement de déchets dangereux de l'Est du Canada et identifie les contaminants préoccupants. Dans trois des sites, la contamination est attribuable à la présence de liquides denses à phase non aqueuse; ces mêmes liquides pourraient être également la cause de la contamination dans les deux autres sites. Ces liquides se sont partiellement dissous et ont formé des panaches de substances chimiques contenant les contaminants préoccupants, p. ex., des composés aliphatiques halogénés, divers composés aromatiques et des BPC. Les méthodes d'échantillonnage sur le terrain, de préservation et d'analyse des échantillons pour ces eaux souterraines polluées sont décrites dans le rapport. L'utilisation d'un spectromètre de masse couplé à un chromatographe en phase gazeuse (GC-MS) pour l'analyse organique quantitative des polluants contenus dans les eaux souterraines est décrite et le problème particulier que pose l'analyse des échantillons fortement pollués fait l'objet d'une discussion. Les principaux aspects du programme d'assurance de qualité/contrôle de qualité de INRE pour les eaux souterraines fortement polluées sont expliqués, en insistant sur l'importance d'obtenir de données très précises et très exactes malgré l'utilisation nécessaire de matrices très complexes.

## MANAGEMENT PERSPECTIVE

During the past year, members of the Ground Water Contamination Project of the Rivers Research Branch of NWRI have undertaken continuing assessments of five hazardous waste sites in eastern Canada. These are the Gloucester Landfill, the Smithville PCB disposal site and the Uniroyal chemical plant site in Ontario; the Ville Mercier dumpsite in Quebec, and the Sydney Steel Works coke oven plant site in Nova Scotia.

The Sydney, Smithville and Mercier sites are considered the three most serious cases of ground water contamination in Canada by the Waterloo Centre for Groundwater Research. All three sites have produced ground water contamination plumes that have polluted former drinking water supplies that now been replaced. All five sites have multimillion dollar clean-up estimates, i.e., from M\$6 for Gloucester to about M\$100 for Smithville. At Mercier, already M\$7 has been spent in an unsuccessful clean-up program that has failed because of the occurrence of dense, non-aqueous phase liquids (DNAPLs) in the subsurface.

The paper describes the procedures required for sampling and analysis of ground water samples at sites with DNAPLs and is the first of its kind in Canada. Ranges of contaminant values are presented for the five sites; all data cited is in the public domain.

NWRI's involvement in the sites arises out of supervision of unsolicited proposals (Mercier, Elmira), responsibility for Federal lands (Gloucester), investment of Federal funds (Sydney) and from a request by Ontario MOE to R.E. Jackson to be a member of a site assessment and remediation review panel (Smithville).

## PERSPECTIVE DE GESTION

L'an dernier, les membres du projet sur la contamination des eaux souterraines de la Direction de la recherche sur les cours d'eau de INRE ont entrepris des évaluations continues de cinq sites d'enfouissement des déchets dangereux de l'Est du Canada : la décharge contrôlée de Gloucester, le site d'enfouissement des BPC de Smithville et la discharge de l'usine chimique Uniroyal, en Ontario; le dépotoir de la ville de Mercier au Québec; et le site d'enfouissement des déchets des fours à coke de la Sydney Steel Works en Nouvelle-Écosse.

Les sites de Sydney, de Smithville et de Mercier sont considérés comme les trois pires cas de contamination des eaux souterraines au Canada par le Centre de recherche sur les eaux souterraines de Waterloo. Ces trois sites ont produit des panaches de contamination des eaux souterraines qui ont pollué les approvisionnements en eau potable qui ont aujourd'hui été remplacés. Le nettoyage des cinq sites coûteraient plusieurs millions de dollars, allant de 6 millions de dollars pour le site Gloucester à environ 100 millions de dollars pour celui de Smithville. À Mercier, 7 millions de dollars ont déjà été dépensés dans le cadre d'un programme de nettoyage qui a échoué à cause de la présence de liquides denses à phase non gazeuse sous la surface.

Ce rapport décrit les méthodes qui doivent être utilisées pour l'échantillonnage et l'analyse des échantillons d'eaux souterraines dans les sites qui contiennent des liquides denses à phase non gazeuse. Il s'agit du premier rapport du genre au Canada. Les variations des valeurs des contaminants sont présentées pour les cinq sites; toutes les données citées sont du domaine public.

La participation de l'INRE à l'étude de ces sites découle de la supervision des propositions spontanées (Mercier, Elmira), de sa responsabilité en matière de terres fédérales (Gloucester), d'un investissement du fonds fédéral (Sydney) et d'une demande du ministère ontarien de l'Environnement à R.E. Jackson en vue de devenir membre d'une commission d'évaluation du site et de révision des mesures d'atténuation (Smithville).

## INTRODUCTION

This paper is concerned with the sampling and analytical methods used at four hazardous waste sites in eastern Canada where contaminated ground waters were discovered. Information from a fifth site, at Smithville, Ontario, has been obtained from information provided by the Ontario Ministry of the Environment. The locations of the sites are shown in Figure 1 and the nature of the contaminant sources is given in Table 1. Two of these sites - Elmira and Gloucester, Ontario - are situated on outwash sand and gravel aquifers. Two more - Sydney, Nova Scotia and Smithville, Ontario - are situated on shallow, fractured sedimentary bedrock. The fifth site at Mercier, Quebec, is underlain by an esker which is in turn underlain by fractured bedrock. All sites, with the possible exception of that Sydney, have produced organic contaminant plumes that threaten ground-water supplies now or formerly used as drinking water sources (see Table 1).

## GROUND-WATER QUALITY AT HAZARDOUS WASTE SITES

It is becoming increasingly apparent that ground-water quality at most hazardous waste sites is dominated by the occurrence of light and/or dense non-aqueous phase liquids, i.e., LNAPLs or DNAPLs. DNAPLs have been identified in ground-water samples and by fouled downhole equipment at three of the five hazardous waste sites listed in Table 1; their presence in the other two sites is suspected but not

confirmed. In addition to DNAPLs, samples from monitoring wells at Sydney, Nova Scotia, contained LNAPL, i.e., lighter than water non-aqueous phase liquids. Ground waters migrating around NAPL pools or through parts of aquifers containing residual NAPL emulsions, i.e., droplets or "ganglia", will become contaminated by mass transfer of the NAPLs to the ground-water. As Hunt et al. (1988) have shown, such mass transfer is very slow and dependent upon the solubility and molecular diffusivity of the organic compounds comprising the NAPL, the rate of ground water flow past the NAPL zones and the surface area of the NAPL. The process by which a sinking DNAPL plume dissolves and contaminates a whole aquifer is shown in Figure 2 from Feenstra and Cherry (1988).

The concentration of the "i"th component of the NAPL in ground water is given by the approximation (Stumm and Morgan, 1981; Bannerjee, 1984):

$$C_i = x_i C_s$$

where  $x_i$  is the mole fraction of the "i"th component in the DNAPL and  $C_s$  is the aqueous solubility of the component. Two important observations may be made about this expression.

First, the equilibrium concentration of the "i"th contaminant in ground-water adjacent to an NAPL pool will be less than the aqueous solubility limit by a factor proportional to the mole fraction of the contaminant in the pool. This is because the contaminant preferentially partitions into the pool (Reinhard et al., 1984). Second, in



the case of several contaminants being present in an NAPL pool with similar mole fractions, the concentration of each in adjacent ground-water will be a function of their respective aqueous solubilities. With time, the more soluble components will preferentially dissolve in the ground-water because such mass transfer is primarily a function of aqueous solubility rather than ground-water flow rate (Anderson et al., 1987). This phenomenon is observed in the weathering of oil seeps (Hunt, 1979) and in the remediation of solvent plumes in ground-water (Feenstra, S., personal communication).

Volatile halogenated aliphatics, pesticides and aromatic hydrocarbons, have dissolved in ground-water from NAPL pools and/or ganglia at the sites listed in Table 1 and have created plumes with aqueous concentrations of up to tens of mg/L. Because these concentrations exceed drinking water limits by as much as one thousand fold, the presence of these contaminants is of foremost concern.

#### SAMPLING OF GROUND-WATER QUALITY AT HAZARDOUS WASTE SITES

The sampling of such ground waters requires a careful choice of monitoring instruments. The principal criterion of choice being that individual hydrostratigraphic units within a ground-water flow system must be individually sampled. Wells that penetrate more than a single unit provide little useful information (Grisak et al., 1978; Reilly et al., 1989). This is partly because the sample dilution that occurs in fully penetrating wells implies dispersion greater than

takes place and also because this integrated sample may well indicate contaminant concentrations within the acceptable guidelines, although these guidelines may be exceeded within a particular unit.

Figure 3 shows two commonly used devices for sampling ground water quality - the bundle-type multilevel sampler and the 5 cm i.d. piezometer. Both provide the capability of sampling small zones of potentially-contaminated ground water. The first is frequently used to map the outlines of contaminant plumes in three-dimensional detail (e.g., Jackson et al., 1985). The second is generally used for monitoring ground-water quality where it is necessary to establish that this quality is in compliance with regulated or guideline values, consequently the well screen is made of an inert material, e.g., stainless steel.

At the Gloucester Landfill, samples for compliance monitoring are collected using dedicated, submersible pumps (QED Systems, Ann Arbor, Michigan) with PTFE (Teflon) bladders operated by compressed air or nitrogen which does not come into contact with the ground-water sample. The pumps, shown in Figure 3, are located at the depth of the well screen and can be isolated from the stagnant water in the well bore by inflating a packer system immediately above the pump. Generally, at least three well screen volumes are then pumped before sampling begins (Barcelona and Helfrich, 1986; Robin and Gillham, 1987).

Figure 4 shows the sequence of operations conducted in the field to collect samples and the subsequent distribution of aliquots for analytical purposes. Samples are collected in precombusted amber glass bottles, with no headspace for volatile organic samples, at a delivery rate of 100 mL/min or less. They are allowed to overflow bottles by at least 1.5 volumes then rapidly capped and stored at about 4°C until analysed (Barcelona et al., 1985). Sample bottles should not be rinsed out with the sample because of the potential for films of NAPLs to coat the bottles.

Preservation techniques for organic constituents of ground-water differ from those for inorganics. In particular, it is inadvisable to subject samples collected for volatile organic analysis to vacuum filtration because of the potential volatilization of analytes. However, samples for analysis of volatile aromatic hydrocarbons, e.g., BTEX in Table 1, should be preserved with HCl acid (U.S. EPA, 1984).

#### ANALYTICAL CHEMISTRY OF ORGANIC POLLUTANTS

The contaminants of concern at the five sites in Table 1 were all organic, consequently their analysis at NWRI was by coupled gas chromatography-mass spectrometry (GC-MS). The principal components of GC-MS systems are shown in Figure 5. Essentially, the ground-water sample containing a mixture of chemicals was first concentrated (purge and trap) and then separated into individual chemicals in the capillary column of the GC, each compound yielding a peak on the chromato-

graph indicating a different retention time within the GC column. The chemicals elute from the GC and are then ionized into fragments of characteristic mass producing mass spectra that allow the identification of each chemical. It should be noted that GC alone cannot confirm the identity of a chemical because many chemicals have similar retention times in GC columns, consequently MS is an essential step in compound identification.

GC-MS analysis may be done in two modes - full scan or selected ion monitoring (SIM). In the first case, a range of masses (typically 45 to 450 atomic mass units) is acquired at the rate of one scan per second, i.e., every second a full mass spectrum is obtained and stored in the GC-MS's dedicated computer for later interpretation. In the second case, only those ions characteristic of the contaminants of concern are acquired, therefore SIM maximizes the sensitivity and selectivity of the GC-MS system. However, if a large number of analytes are requested by the hydrogeologist, e.g., "a volatile organic scan", then these gains are offset by the flexibility of the full scan mode. Because only a few masses are acquired by SIM, it is not possible to identify unknowns in the sample using SIM, furthermore, in heavily contaminated samples, interferences may cause misidentification and bias the quantitation. Figure 6 shows the full scan or total ion current chromatogram for volatile organics in a sample of ground-water from the Gloucester landfill, while Figure 7 shows a SIM chromatogram for four selected ions.

Even when a full spectrum is acquired, quantitative analysis is done on extracted ions. One ion is selected for each compound for quantitation, i.e., each ion is a specific detector. The area under the curve of each peak of the chromatogram is integrated and compared to that of a standard. In addition, the area of one or two other characteristic ions is also integrated and their ratios compared to that of the primary ion; these are known as qualifying ions because they allow qualitative identification of the compound. Under the same operating conditions of the GC-MS, the ratios of qualifying to primary ions are constant. These, together with the retention times of the compounds in the GC column, comprise the criteria used to ensure the correct identification of the analytes.

The identification of the compounds contained within a sample is accomplished by comparing the spectrum of the unknown to spectra contained within a library stored within the GC-MS computer. There are two methods of comparison - forward and reverse searches. In a forward search, the spectrum of the unknown is compared to those contained in a library, e.g., that of the U.S. National Bureau of Standards or a user-prepared library. The search yields a list of matches ranked in order of best fit. It is not uncommon to have matches of 90-95%, which would be considered confirmatory. However, when the matches are much less, the chemist must interpret the differences and make tentative identifications. In a reverse search, a group of spectra contained in a user-created library is compared to all the spectra found in the sample. This type of search is commonly used in target compound or "priority-pollutant" analysis.

Therefore, whenever a hydrogeologist requests a "GC-MS scan" of a laboratory, it is essential that he or she specify the method of searching. Forward searches will also identify non-priority pollutants that may be of considerable value in identifying the origin or pathway of the contaminated ground-water (Swallow et al., 1988), however, they are more time consuming and therefore more costly than reverse searches. The latter have the advantage of being able to identify much lower quantities of a compound than is possible in a forward search.

Consequently, the advantages of the mass spectrometer as a detector over other detectors (ECD, FID, etc.) are that the quantitation is not affected by coeluting compounds and that the spectrum allows the unambiguous identification of the target compounds and tentative identification of non-target compounds.

#### QUALITY ASSURANCE/QUALITY CONTROL

The purpose of any quality assurance (QA) program is to reduce analytical measurement errors to agreed upon limits (quality control or QC) and to ensure that the analytical results have a high probability of being of an acceptable quality, a system known as quality assessment (American Chemical Society, 1983).

QA/QC programs need to be operated at two different levels. The first is within the laboratory to ensure that good lab practice is continually employed and that the results achieved are of consistent

and acceptable quality. Secondly, external control by the client has to be implemented to ensure comparability of data obtained from different sources and over different time periods.

Two principal factors need to be assessed by the QA/QC program - precision and accuracy. They are defined as follows:

Precision: The degree to which data generated from replicate or repetitive measurements differ from one another (ACS, 1983).

Accuracy: The degree of agreement of a measured value with the true or expected value of the quantity of concern (Taylor, 1987).

Any reputable commercial laboratory will provide a hydrogeologist with information on the precision of their methods, however, it is the responsibility of him or her to state the required number of replicates necessary to assess the validity of the method for their own samples and the laboratory performance. Accuracy is more difficult to assess because, in many cases, certified reference materials, in particular for volatile organic compounds, are not available for many analytes and sample matrices (i.e., the totality of the sample, e.g., landfill leachate) and, furthermore, interlaboratory studies are infrequent. A suitable alternative would be to have another laboratory prepare a set of standards and spiked samples to be included with the rest of the samples. It should be stressed that split samples give little or no information about either precision or accuracy.

The principal QA/QC tools used by NWRI in its assessment of the sites shown in Table 1 were the employment of blanks, replicates, surrogates and internal standards. As a rule, field or trip blanks were collected to evaluate external contamination of the sample, e.g., benzene from gasoline stored near the sample bottles, and the results corrected on the basis of such blanks. Replicates are important because they are the only measure one has of sample variability. Therefore, it is preferable to analyse one sample in triplicate and thus obtain statistics on the variability than to analyse two samples in duplicate, for which statistics cannot be estimated.

Surrogates, compounds that are analogs of the analytes and which are added to the sample prior to the extraction step, have replaced spiked samples as QA/QC tools. This is because the latter are impractical in that the laboratory does not usually know the expected concentration of the analyte a priori, therefore cannot spike the sample to a meaningful level. Most surrogates are deuterated (deuterium isotope labelled) analogs of the analyte of interest or chemical analogs, e.g., bromodichloromethane for volatile organics, assumed unlikely to be present in the sample. Since the surrogate is added prior to extraction, it is carried through the entire analytical procedure and its recovery reported as a measure of matrix effect and analytical error.

The final QA/QC tool used at NWRI was that of internal standards. These too may be deuterated analogs, however, they are added in the final step and are used for quantitation. Their purpose



is to account for instrument variability from one sample to the next. Thus the peak area of the internal standard in the sample chromatogram is compared to that in the standardization chromatogram and all concentrations are corrected for any variability.

#### THE ANALYSIS OF CONTAMINANTS OF CONCERN IN HIGHLY CONTAMINATED GROUND WATERS

In most of the cases shown in Table 1, the concentration of one or more analyte exceeded the working range of the method of analysis, and it was necessary to dilute the sample to within the working range. This naturally results in a corresponding reduction in the detection limit of all the analytes present in the sample. Indeed, minor constituents will be lost. Attempts to analyse both diluted and undiluted samples are often frustrated by analyte carryover in the GC-MS, i.e., the release of sorbed analyte from the instrument itself. While it is sometimes possible to isolate semi-volatile compounds, e.g., PAHs and PCBs by liquid chromatography, this approach will not work for volatile organics which comprise a large part of the contaminants of concern in the ground waters in Table 1. It is thus customary to dilute highly-contaminated samples with "organic-free" water, i.e., water of extremely low dissolved organic carbon.

In such a case, caution must be exercised in evaluating results. For example, when a sample has been diluted one hundred fold, analytical error and detection limit should be multiplied by one hundred. If

the blank contains a few compounds close to the detection limit, they should also be multiplied, e.g., a residual concentration of 0.3  $\mu\text{g}$  benzene/L will be reported as 30  $\mu\text{g}$  benzene/L, which may seem significant. However, in this case the detection limit should be reported as 10  $\mu\text{g}/\text{L}$ , and not 0.1  $\mu\text{g}/\text{L}$  for the undiluted sample. It would be best, in reflecting the actual precision of the data, to change units and report the sample value as 0.03 mg/L and the detection limit as 0.01 mg/L.

#### SUMMARY AND CONCLUSIONS

NAPLs, both light and dense, are the primary cause of ground-water contamination at the three hazardous waste sites studied in Eastern Canada, and may well be the cause at two others. The plumes of ground-water contamination produced by them require careful sampling and analytical procedures to allow hydrogeologists to assess the nature of each problem. Trace analytical techniques developed for very low organic concentrations in natural waters cannot always be used in such heavily polluted ground waters.

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## LIST OF CAPTIONS

Figure 1. The location of the five hazardous waste sites in Eastern Canada.

Figure 2. Ground-water contamination from residual DNAPL and DNAPL pools (from Feenstra and Cherry, 1988).

Figure 3. Types of monitoring instruments: (a) the bundle-type multilevel and (b) the 5 cm i.d. piezometer with a dedicated submersible pump.

Figure 4. Flow chart of sampling and analysis methods used by NWRI.

Figure 5. Components of a GC-MS system (from Swallow et al., 1988).

Figure 6. Total ion current chromatograms: (a) schematic and (b) TIC for multilevel sampling point 67M-11 at Gloucester.

Figure 7. Selected ion monitoring.

Table 1. Concentration ranges of contaminants of concern in ground waters at five hazardous waste sites in Eastern Canada. ppm (>1 mg/L), ppb (= 1-1000 µg/L) and ppq (= pg/L) indicate maximum levels detected in several monitoring wells at each site.

Site	DNAPL composition	Contaminants of Concern in ground-water plumes
Elmira	presence uncertain	BTEX, phenols and CPs (ppm); 2,4-D and 2,4,5-T (ppb), PCDD (soil)
Gloucester	presence uncertain	F113, solvents, VC, dioxane, benzene (ppb); PCDDs and PCDFs (ppq)
Mercier	oil + solvents?	solvents, pesticides, benzene, CB and VC (ppm)
Smithville	oil + TCE + PCB + CBs	PCBs, TCE and CBs (ppb)
Sydney	coal tar	BTEX and naphthalene (ppm)

Abbreviations:

BTEX = benzene, toluene, ethyl benzene and xylene  
 CBs = chlorobenzenes  
 CPs = chlorophenols  
 dioxane = 1,4-dioxane  
 F113 = Freon-113 (1,1,2-trichloro-1,2,2-trifluoroethane)  
 PCB = polychlorinated biphenyls  
 PCDD = polychlorinated dibenzodioxins  
 PCDF = polychlorinated dibenzofurans  
 solvents = tetrachloroethene (PCE), trichloroethene (TCE), 1,1,1-trichloroethane (TCA), 1,2-dichloroethane  
 VC = chloroethene (vinyl chloride)  
 2,4-D = 2,4-dichlorophenoxyacetic acid  
 2,4,5-T = 2,4,5-trichlorophenoxyacetic acid

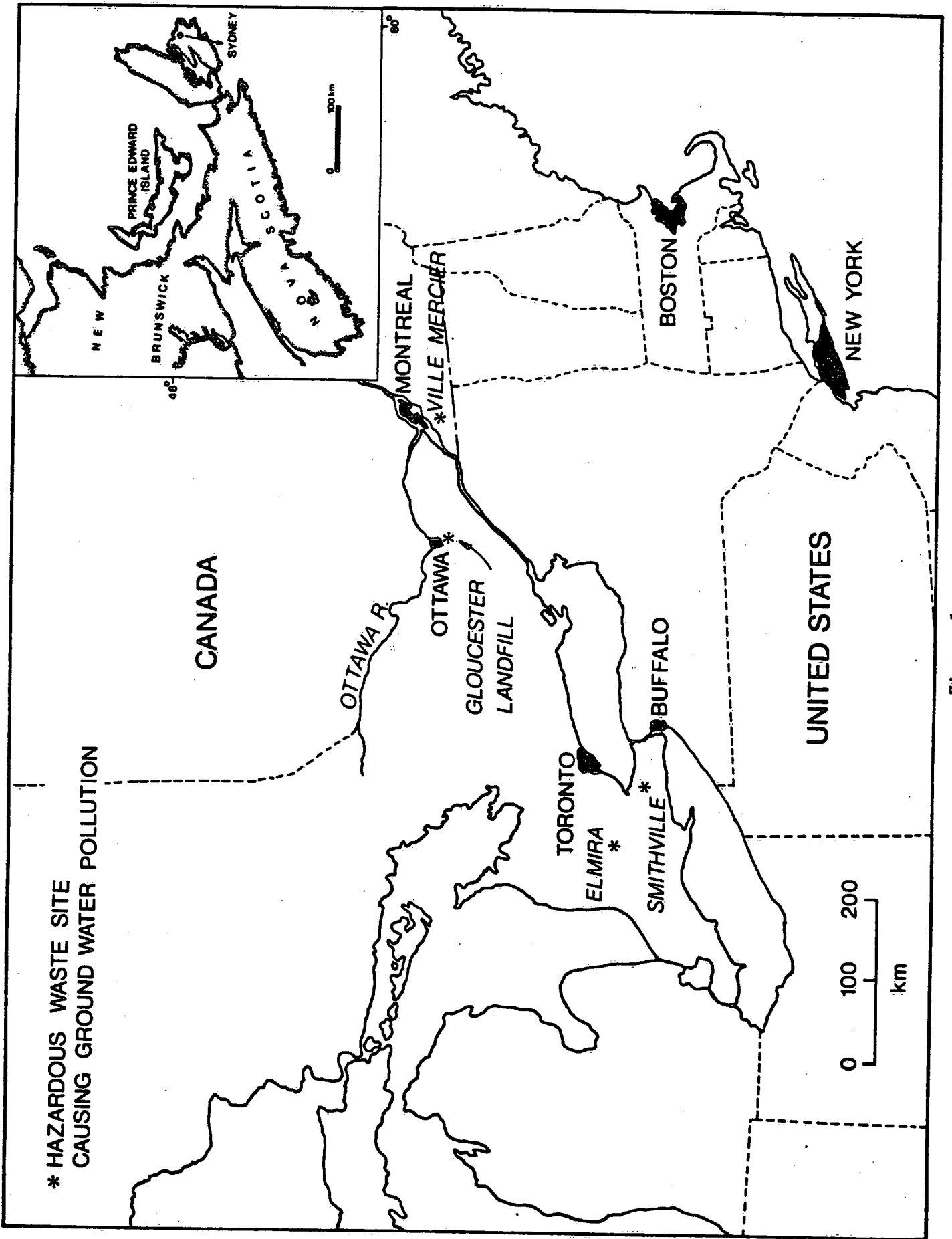


Figure 1

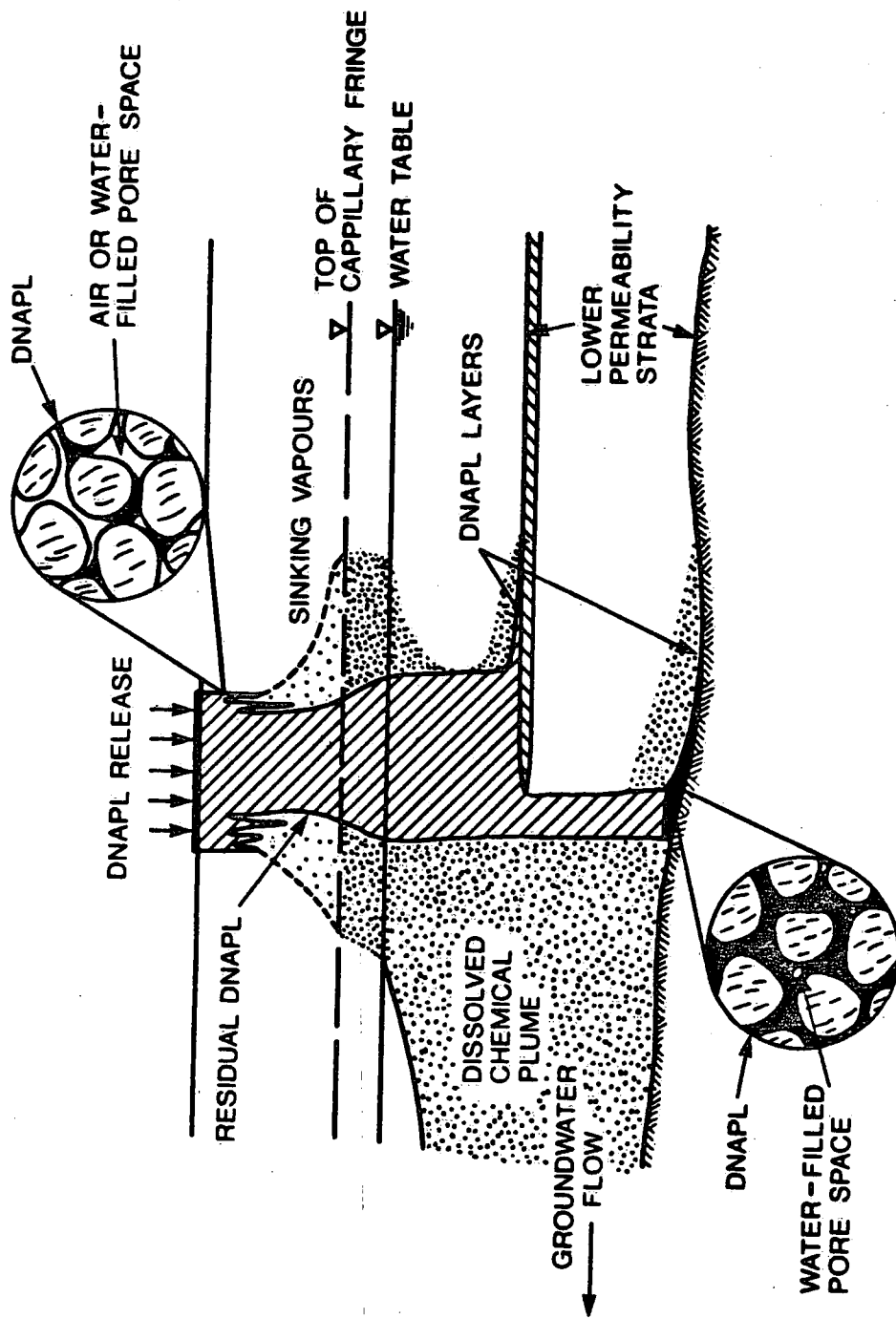


Figure 2



**MULTILEVEL SAMPLER**

**MONITORING WELL (124P-128P, 134P)**

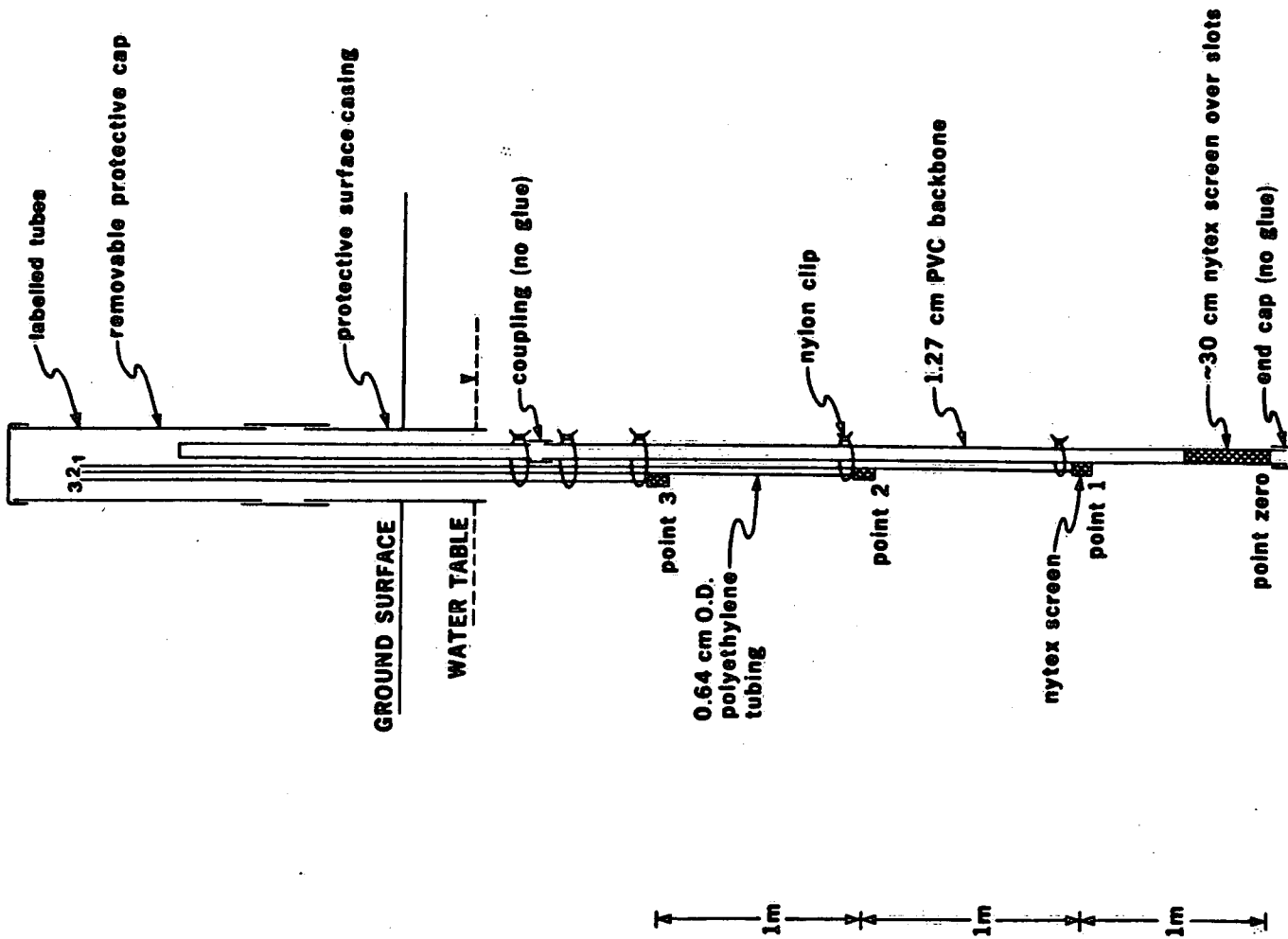
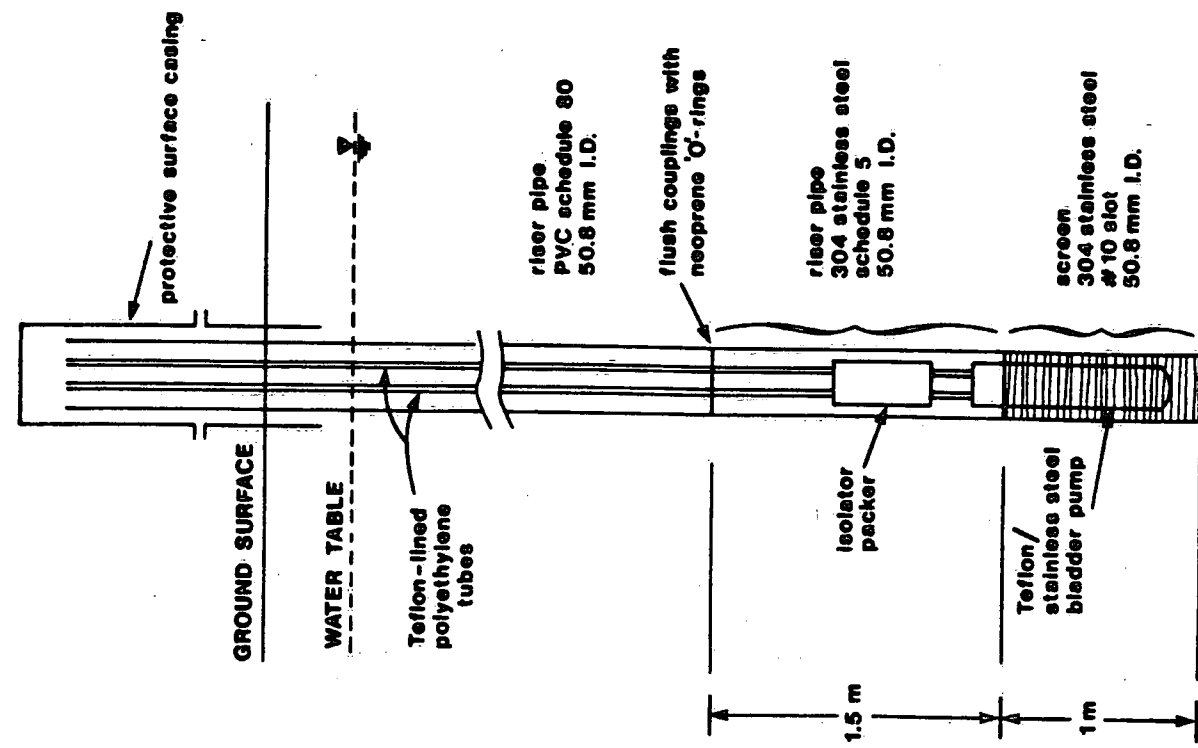


Figure 3

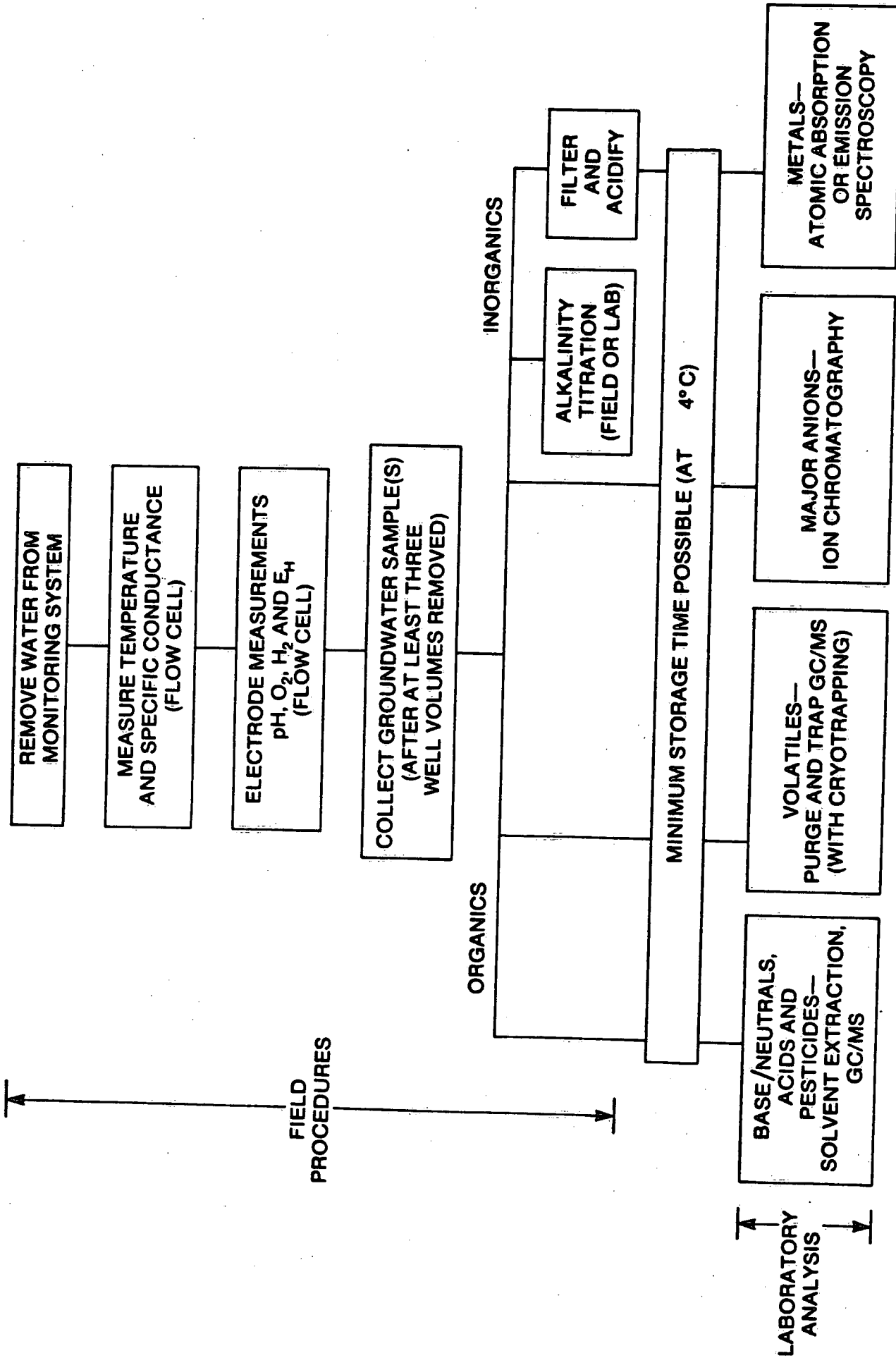
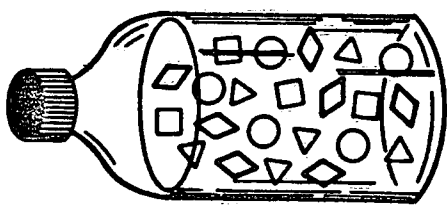


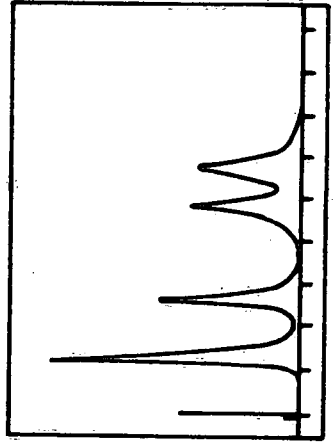
Figure 4

COMPONENTS OF A GC/MS SYSTEM

SAMPLE



GAS CHROMATOGRAPHY



MASS SPECTROMETRY

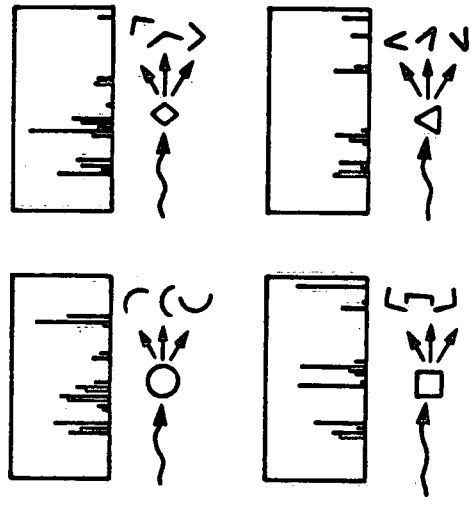
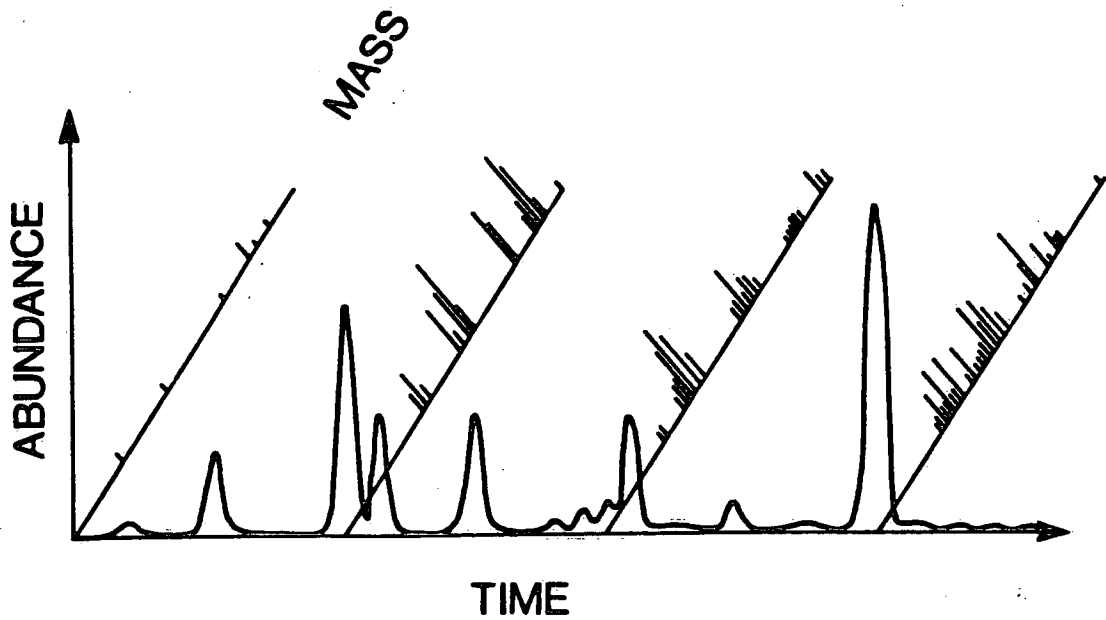


Figure 5



TOTAL ION CHROMATOGRAM

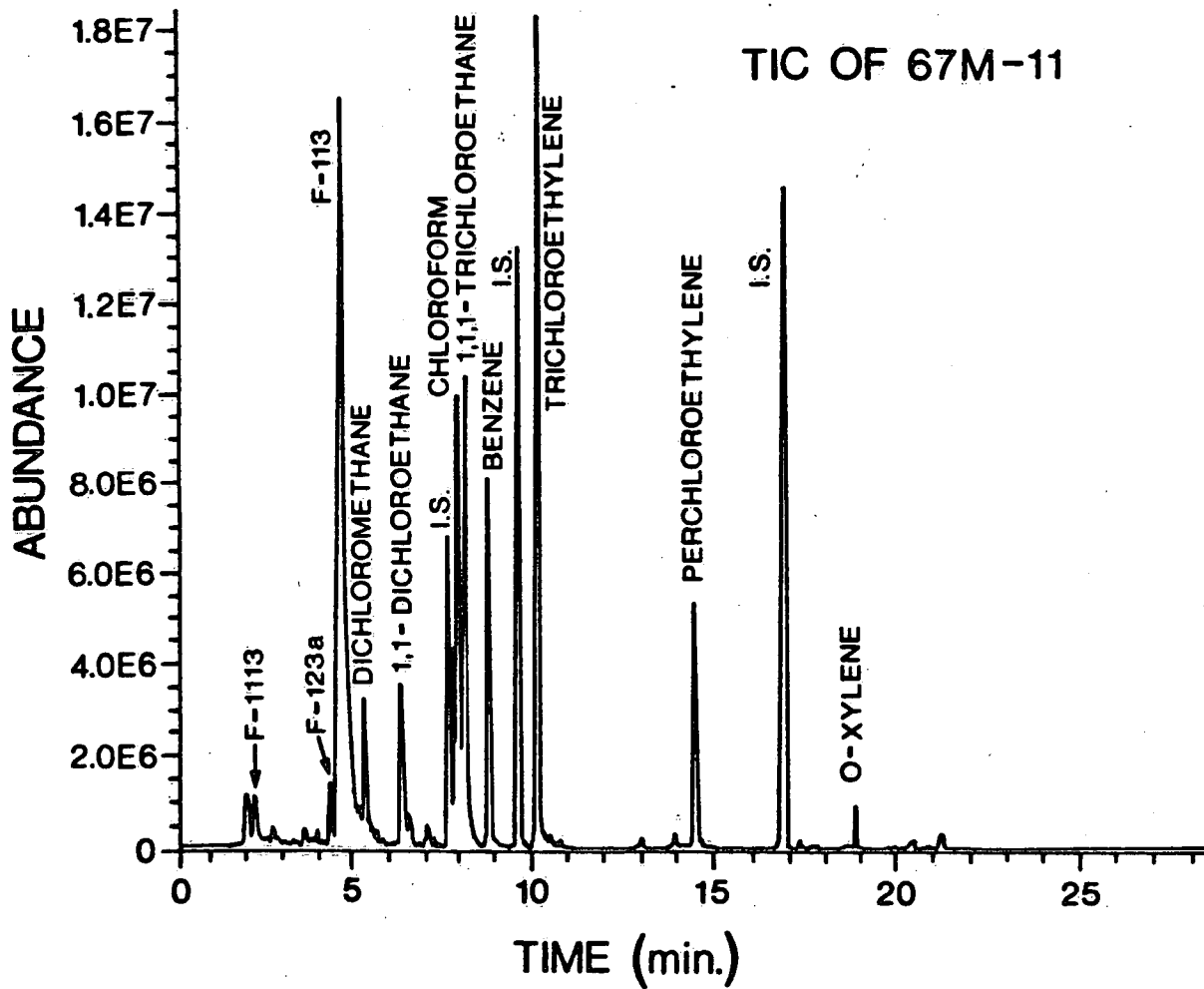
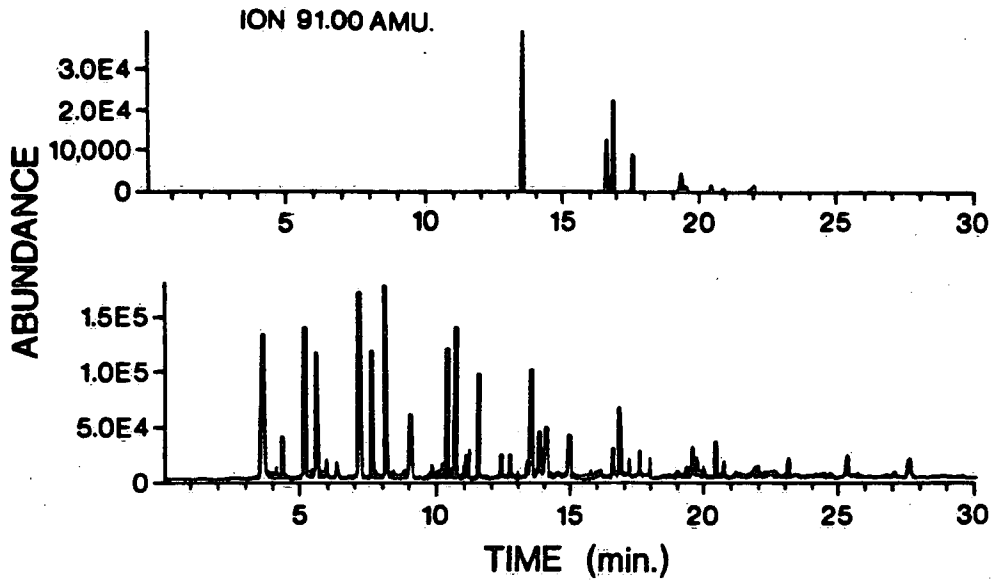


Figure 6

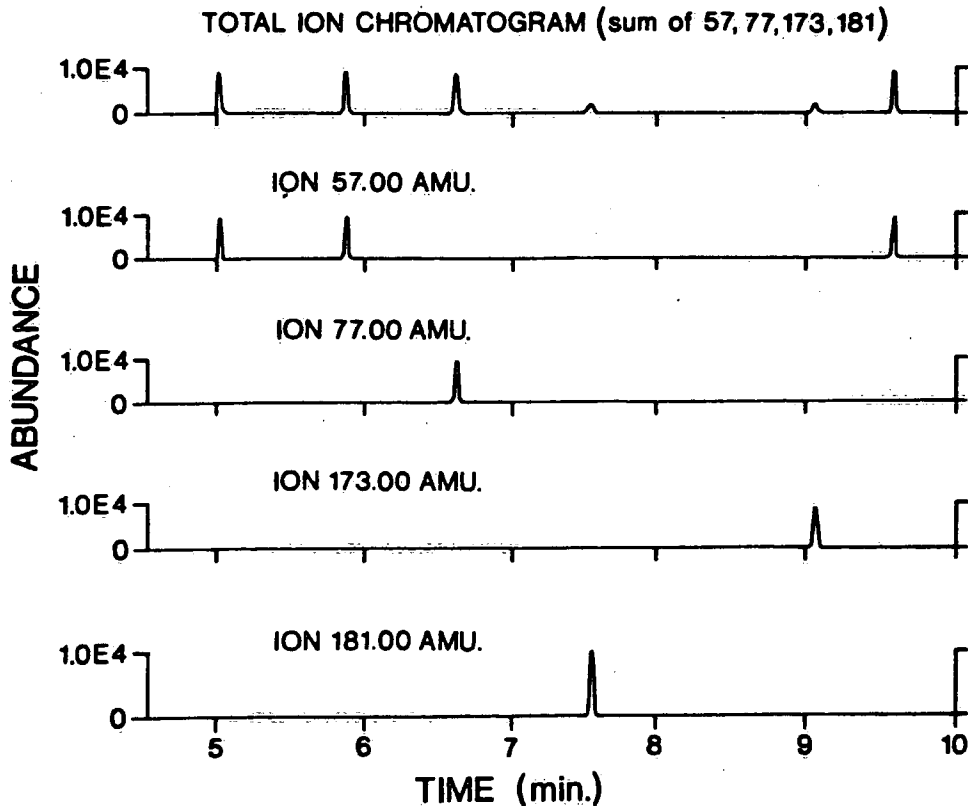
### (a) EXTRACTED ION CHROMATOGRAM

A RANGE OF IONS (e.g. 45 to 450 a.m.u.)  
IS ACQUIRED, BUT SELECTED IONS ARE QUANTITATED



### (b) SELECTED ION MONITORING

- ONLY SELECTED IONS ARE ACQUIRED
- MAXIMIZES SENSITIVITY AND SELECTIVITY
- DOES NOT ALLOW THE IDENTIFICATION OF UNKNOWNNS



EACH CHANNEL IS A SPECIFIC DETECTOR

Figure 7