



Project Summary

Development of a Capillary Wick Unsaturated Zone Pore Water Sampler

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Existing unsaturated zone soil water samplers have several deficiencies which jeopardize their utility for field sampling. Suction cups only function when a vacuum is applied, and sample from an unknown volume of soil. Pan samplers only sample saturated flow. A capillary wick sampler was developed to overcome these problems. Materials for its construction were selected and tested for conductivity, capillary potential and chemical inertness. Breakthrough curves for selected inorganic ions and organic chemicals were established in the laboratory. No adsorption/desorption of these chemicals was found for the capillary wick sampler, the suction cup sampler, or the pan sampler. Banks of 8 capillary wick samplers were installed in test plots of undisturbed soils having sand, silt loam and clay textures. Bromide breakthrough curves were determined at each location. The data were used to determine the number of samplers required to characterize the flow of contaminants resulting from a uniform application to the soil surface. These results indicated that to achieve 95% confidence, 31 samplers would be required in the sandy soil, 6 in the silt loam soil and 2 in the clay soil.

The experimental plots were drained and samples were collected over a range of soil moisture contents and soil moisture potentials. It was demonstrated that the

wick sampler does adequately collect soil solution samples from soils having soil moisture potentials ranging from 0 to -60×10^{-4} MPa. The capillary wick sampler is an improvement over existing samplers since it does not require continuous suction to provide continuous samples and because it can collect samples of flow which takes place when the soil is unsaturated. While the sampler collects volumes representative of the flux at potentials of -50×10^{-4} MPa, convergence at greater potentials and divergence at lower potentials prevent its use as a tool for measuring flux of water or contaminants.

This Project Summary was developed by EPA's Environmental Monitoring Systems Laboratory, Las Vegas, NV, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Samples of water moving through the unsaturated zone between the soil surface and the groundwater table are needed to detect and monitor any mobile contaminants which may be moving toward the groundwater. Soil water samples collected from the unsaturated zone could provide early warning of potential groundwater pollution arising from agricultural activities such as fertilizers, pesticides, and salts; industrial activities such as hazardous waste piles, landfills, pits, ponds, and lagoons; and

commercial activities such as buried fuel storage tanks.

Present techniques for unsaturated zone pore water sampling consist of suction cup or pan (free drainage) samplers (EPA 1977).

Each of these sampling techniques have several flaws which make them difficult to use and cast uncertainty on the validity of the collected samples. This project was, therefore, undertaken to develop an improved unsaturated zone soil water sampler which would overcome some, if not all, the shortcomings of the present samplers.

Methods and Materials

Laboratory Study Procedures

A laboratory study was conducted to document the ability of three types of samplers to sample known concentrations of organic and inorganic chemicals. To accomplish this, three samplers of each type; suction cup samplers, glass block pan samplers, and capillary wick samplers were tested by exposing them sequentially to distilled deionized water, water with a known concentration of organic or inorganic chemical, and finally distilled deionized

water again. The first solution contained 200 mg Cd L⁻¹ and 220 mg NO₃ L⁻¹, added as CdNO₃. The second test solution contained 100 mg Br L⁻¹, added as KBr. The third solution contained 13.4 mg L⁻¹ trichloroethylene, 40.0 mg L⁻¹ toluene, 9.9 mg L⁻¹ ethylbenzene, and 13.6 mg L⁻¹ naphthalene. Samples of the effluent containing organics were placed in 22 mL headspace vials sealed with a Teflon coated silicone rubber septum and aluminum crimp cap and stored on ice until analysis. Because the physical size and shape of the various lysimeters differed greatly, it was necessary to normalize the data. This was done by dividing the volume of collected sample by the surface area of the sampler to obtain what will be hereafter referred to as an equivalent depth. Sampling intervals were equivalent to 0.1, 0.2, 0.3, and 0.5 cm equivalent depth for the background distilled deionized water and 0.1, 0.2, 0.3, 0.5, and 1.0 cm equivalent depth for the test solution and the following distilled deionized water.

Capillary Wick Sampler Design

Three capillary wick samplers were constructed, as shown in Figure 1. Materials used to construct the capillary

wick sampler were chosen for (1); the non-reactivity to inorganic and organic compounds and (2); the absence of substrate for microbial growth. Five centimeter diameter Pyrex tubing was epoxied to the glass plate. Additional sections of pyrex tubing were attached to one another using teflon lined gaskets. A 5 liter pyrex bottle was used as a sample collection chamber. A 1.27 cm diameter glass wicking was chosen for use in this study due to its capillary rise of 54 cm and high saturated conductivity of 10⁻² cm sec⁻¹. The woven glass cloth used to construct the sampler to cover the exposed wick.

Sample Analysis

Bromide and nitrate analyses were done using specific ion electrodes (Orion No 94-35A and 93-07, respectively). Cadmium analyses were done using Perkin Elmer model 603 Atomic Absorption Spectrophotometer and an acetylene flame.

Organic analyses were done using Hewlett-Packard (HP) 19395A Headspace Sampler coupled to an HP gas chromatograph (GC) model 5890, which was in turn coupled to an HP mass selective detector (MSD) model 5970. The HP high performance (SE-52) cross

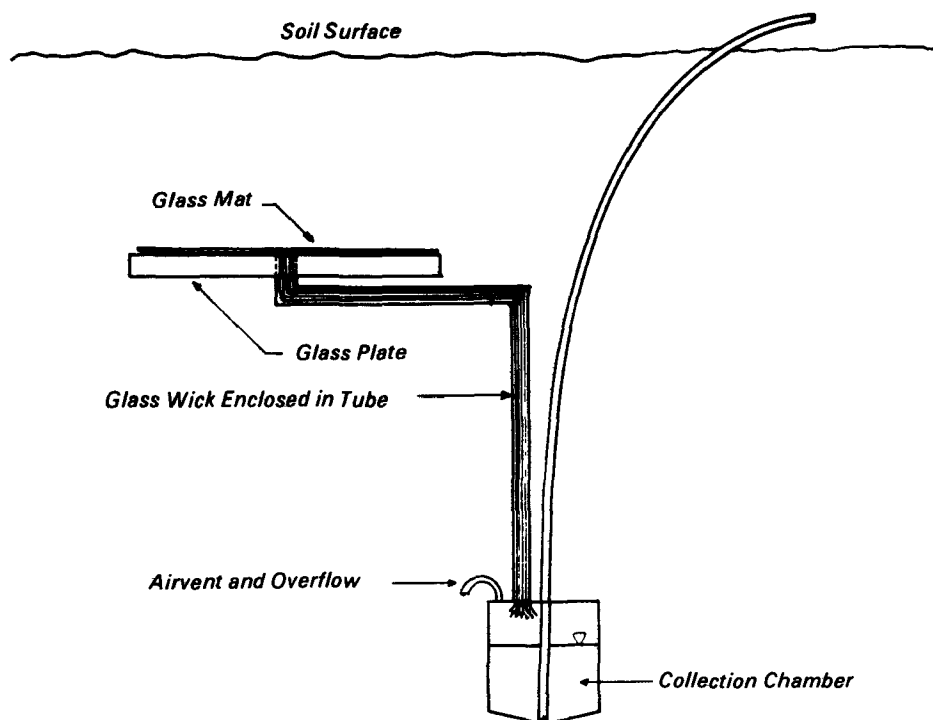


Figure 1. Schematic diagram of the unsaturated zone capillary wick sampler.

linked 5% phenyl methyl capillary column with a film thickness of 0.11 μm , an internal diameter of 0.20 mm, and a length of 25 m was used for all organic analyses. The tuning calibration compound used was perfluorotributylamine (PFTBA).

Field Study Procedures

Sampler Construction

Glass plates, 0.96 cm thick were purchased and cut to 30 cm x 30 cm for use in the Weswood silt loam and the Lufkin clay. Plates 25 cm x 25 cm were used in the Padina sand. For field installation, 5 cm diameter plastic tubing was substituted for the pyrex tubing. Additionally, 3.8 L glass bell jars were substituted for the pyrex sample collection chambers.

Sampler Installation

Eight samplers were installed in each of three soils; the Weswood silt loam, a fine silty, mixed thermic fluventic ustochrept; the Padina sand, a loamy, siliceous, thermic grossarenic paleustalf; and the Lufkin clay, a vertic albaqualf, fine, montmorillonitic thermic using the following procedure.

A plot area 2.4 m by 3.7 m was delineated and a trench 4.3 m long 1.2 m wide and 1.5 m deep was excavated with a backhoe along one long side of the plot area. Four excavations conforming to the dimensions of the sampler were dug horizontally 46 cm into the trench sidewall and samplers were placed into the excavations. The area around and beneath each sampler was then carefully backfilled and compacted to prevent cavities and/or damage to samplers due to soil shifting. The nylon sample removal tubing was encased in 2.54 cm diameter PVC pipe which acted as a conduit to the soil surface.

To facilitate drainage, a 60 cm diameter sump was placed in the center of the trench and surrounded by a 15 cm gravel bed across the bottom of the remainder of the trench. A continuous sheet of plastic was placed against the sampler sidewall and across the top of the gravel bed and the trench was backfilled and compacted. A parallel trench 2.4 m from the sampler sidewall of the initial trench was dug and the procedure repeated. Two end trenches were then dug, filled with a 15 cm layer of gravel, lined with plastic and then refilled and compacted. The resulting plot was a 2.4 m x 3.7 m monolith of undisturbed soil lined with plastic. A set

of gamma probe access tubes (5 cm diameter, 0.9 m deep) were installed to allow measurement of water content. Two sets of tensiometers were installed at depths of 30, 61, and 91 cm. A piezometer was installed to record the water table level. A wooden frame was placed behind the plastic lining and around the plot to a height of 20 cm above the soil surface. A temporary rain shelter was constructed over the plot and water was ponded to a standing depth of 15 cm until the plot became saturated. Upon saturation, a solution containing 100 mg Br⁻ L⁻¹ as KBr was applied to determine Br⁻ breakthrough curves for each sampler. Samples were collected continuously until the Br concentration in the samples equaled that of the applied solution. Bromide measurements were made as previously described in the laboratory section. After breakthrough, the plot was covered with plastic and allowed to drain.

During drainage, soil moisture content and soil moisture potential measurements were made. Water table level and sample volume measurements were made and recorded. Measurements were continued until no sample was collected for a 24 hour time interval. During this time, the soil surface was covered with plastic to prevent upward flux of water. This procedure was repeated 5 times on the Padina sand and 3 times on the Weswood silt loam.

Results and Discussion

Laboratory Study

When expressed on an equivalent depth basis, all samplers achieved breakthrough of inorganic ions by 0.3 cm as shown in Figure 2. None of the samplers had any initial inorganic contaminants, and they did not significantly alter the concentration of any of the added ions. Measurement of the dead volume of the different samplers approximately accounted for the majority of the 0.3 cm equivalent depth required for the change in sample concentration. Most of the maximum concentrations measured were within 5% of that applied and therefore were not considered significant. Therefore, it appears that in reference to the samplers' ability to transmit the tested inorganic ions, none of the samplers has any advantage over the others.

All samplers were free of initial organic contaminants and initial water samples were all below the detection limits for each of the four organics studied (Figure

3). Concentrations of trichloroethylene, toluene, and ethylbenzene rose quickly and were at or very near their maximum by 0.5 cm equivalent depth. Once the application of organics was terminated and clean water was applied, the organic concentrations dropped rapidly and by 0.5 cm equivalent depth were about one tenth of the maximum concentration measured. These data therefore indicate that there is little to no adsorption and desorption of organics on the sampler materials. None of the organic concentrations measured reached the concentrations originally present in the stock solution which was applied probably due to losses to the atmosphere in and around the samplers.

Field Study

Bromide Study

Normalized bromide breakthrough curves for each of the three soils were typical of what was expected. A relative Br concentration of 0.5 was reached 26 to 27 hours after application began to the Padina soil, in 12 to 15 hours for the Weswood soil, and in 113 hours for the Lufkin clay. Variation in bromide concentration collected for each sampler at corresponding times can be attributed to variable flow patterns within the plot and was particularly pronounced in the Padina sand.

The number of samplers required to obtain an accurate estimate of the chemical concentration in each soil texture was determined using the equation based on the 95% confidence limits computed from the sample mean as given by Snedecor and Cochran (1978). Based on this equation, the number of samplers needed for different soils with different accuracy levels are presented below.

Soil	Accuracy Level		
	5%	10%	15%
Number of Samplers			
Sand	31	8	3
Loam	6	2	1
Clay	2	1	1

The number of samplers needed to collect a representative sample in any texture soil decreases as the accuracy level decreases. Variation in number of samplers needed between soil textures at equivalent accuracy levels is due to variation in population standard deviations. The greatest number of samplers needed occurs in the sand soil due to the high standard deviation occurring in that

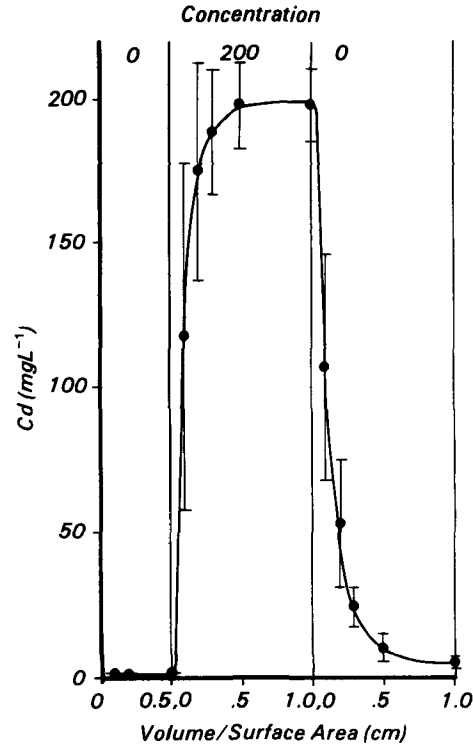


Figure 2. Mean concentration of Cd in samples from pan samplers sequentially exposed to solutions containing 0, 200, and 0 mg Cd L⁻¹.

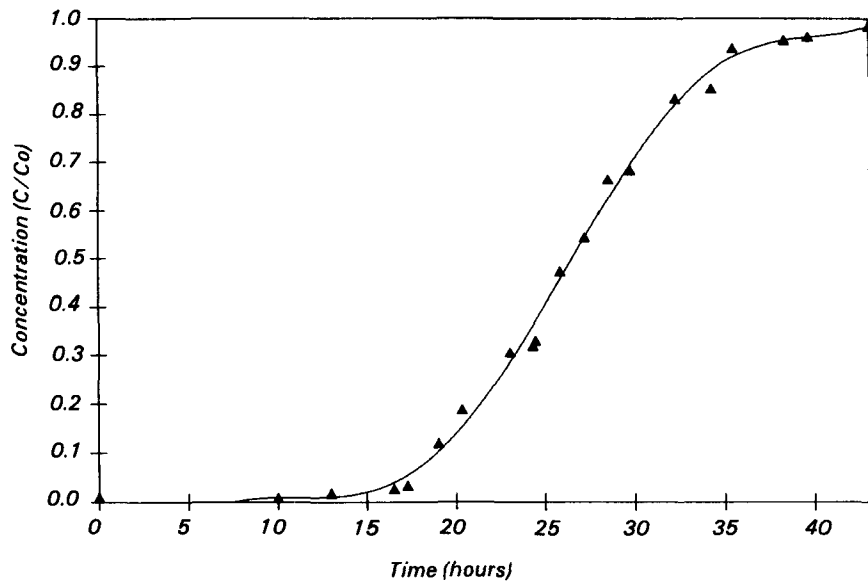


Figure 3. Relative bromide concentration with time in samples from Padina soil. Data points are mean of 7 values.

soil texture. One possible reason for the large population standard deviation in the Padina sand is that there may be numerous irregularities in texture within the plot leading to differential flow patterns in the soil. It may also be possible that saturated flow through macropores in the well structured loam and clay soils was more uniform than flow through the very poorly structured sand. Some of the variability may also be due to the 30% reduction in sampler size needed to accommodate the high saturated conductivity of the sand.

Soil Water Flux Measurements

For the Padina soil, sample collection was near $3,000 \text{ cm}^3 \text{ hr}^{-1}$ at soil moisture potentials near zero after which the volumes decreased rapidly. At soil moisture potentials between -25 to $-85 \times 10^{-4} \text{ MPa}$, the volume collected ranged from 0 to $1360 \text{ cm}^3 \text{ hr}^{-1}$. The collection of samples at potentials drier than $-50 \times 10^{-4} \text{ MPa}$ and the large standard deviations in the Bromide study both indicate the presence of variable flow patterns in this soil. From the data it is apparent that when using these samplers in a sandy soil the sample collection frequency should be determined on a case by case basis, with samples being collected at a frequency sufficient to prevent divergence due to overflow of the collection vessel.

The data from the Weswood were similar in nature and again at wet potentials (0 to $+17 \times 10^{-4} \text{ MPa}$) the sample collection rate ranged from 82 to $4,000 \text{ cm}^3 \text{ hr}^{-1}$. At potentials drier than $-10 \times 10^{-4} \text{ MPa}$ the collection rate ranged from 0.1 to $22 \text{ cm}^3 \text{ hr}^{-1}$. Thus sampling frequency for this soil ought to range from daily at near saturation conditions to weekly at drier soil moisture conditions.

When expressed as fractional flow, it is apparent that the samplers capture up to 11 times the actual soil water flux in the Padina sand at soil moisture potentials from 0 to $-26 \times 10^{-4} \text{ MPa}$. As the soil moisture potential decreases, the fraction of the flow captured crosses one at a potential of $-51 \times 10^{-4} \text{ MPa}$ and decreases to 0 by a potential of $-85 \times$

10^{-4} MPa . A similar trend was seen in the Weswood soil except that the drop was faster.

Advantages of the Capillary Wick Sampler

1. The sampler collects an adequate sample volume for laboratory analysis over a range of soil textures, soil moisture contents, and soil moisture potentials.
2. It is possible to determine the number of samplers needed to obtain a representative sample of soil moisture flux constituents in various soil textures.
3. Once sampler installation is complete, it requires little energy input to a) maintain the samplers and b) to collect a sample.
4. The sampler can collect inorganic ions and organic chemicals without altering the concentration of the sample constituents collected.
5. The sampler is composed of inert materials which will not react with inorganic ions or organic chemicals.
6. The sampler materials should not present a substrate for microbial growth.
7. The sampler provides continuous suction without the use of a vacuum system. Therefore, a sample can be collected from any soil which has a matric potential wetter than -70 cm of water.
8. The sampler provides continuous collection of leachate, therefore periodic removal of samples should provide an integration of the concentration of constituents passing a given depth.
9. The capillary wick sampler is suitable for use in all soil textures. Best results are obtained in well structured soils, i.e., the loam and the clay, as opposed to unstructured soils such as the sand.

Conclusions

A capillary wick sampler capable of maintaining a potential of $-50 \times 10^{-4} \text{ MPa}$ was designed, constructed from commercially available materials, and tested in both the laboratory and field. Laboratory testing of the capillary wick

sampler showed no adsorption/desorption of inorganic ions or organic chemicals. Breakthrough curves of inorganic ions and organic chemicals were similar for the capillary wick sampler, the suction cup sampler and the pan sampler.

Field measurements over a range of soil textures indicated that to collect a sample representative of the chemicals flowing in the soil solution within 95%, 90%, and 85% confidence intervals that sandy soils would require 31, 8, and 3 samplers, loam soils 6, 2 and 1 samplers, and clay soils 2, 1 and 1 samplers, respectively. Use of the capillary wick sampler to measure soil moisture flux is not feasible. When soil moisture contents are high, the sampler collects in excess of 100% of the soil moisture which should be intercepting the glass plate due to convergence of soil moisture in response to the potential generated by the wick material. These data also suggest that the capillary wick sampler should not be used to determine soil moisture flux in areas where a perched water table exists within 50 cm below the glass plate. Soil moisture samples can still be collected but care should be taken in data interpretation since back siphoning from the water table into the sampler may occur. As the soil dries to $-50 \times 10^{-4} \text{ MPa}$, sample volumes collected approach 100% of the soil solution intercepted by the sampler. At potentials drier than $-50 \times 10^{-4} \text{ MPa}$, less than 100% of the soil solution which should be intercepted by the sampler is collected. Divergence of soil moisture flow can also occur if the sampler collection chamber is not emptied before it fills completely.

References

- EPA, 1977. Procedures Manual for Groundwater Monitoring at Solid Waste Disposal Facilities. U.S. EPA Office of Solid Wastes. SW-616.
- Snedecor, G. W. and W. G. Cochran. 1978. Statistical Methods. Iowa State University Press. Ames, Iowa, pp. 516-517.

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Lawrence Eccles is the EPA Project Officer (see below).

The complete report, entitled "Development of a Capillary Wick Unsaturated Zone
Pore Water Sampler" (Order No. PB 89-129 100/AS; Cost: \$21.95, subject to
change) will be available only from:

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