Designation: D 4696 – 92 (Reapproved 2000)

Standard Guide for Pore-Liquid Sampling from the Vadose Zone[1]

This standard is issued under the fixed designation D 4696; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide discusses equipment and procedures used for sampling pore-liquid from the vadose zone (unsaturated zone). The guide is limited to in-situ techniques and does not include soil core collection and extraction methods for obtaining samples.

1.2 The term "pore-liquid" is applicable to any liquid from aqueous pore-liquid to oil. However, all of the samplers described in this guide were designed, and are used to sample aqueous pore-liquids only. The abilities of these samplers to collect other pore-liquids may be quite different than those described.

1.3 Some of the samplers described in this guide are not currently commercially available. These samplers are presented because they may have been available in the past, and may be encountered at sites with established vadose zone monitoring programs. In addition, some of these designs are particularly suited to specific situations. If needed, these samplers could be fabricated.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this guide may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

3. Terminology

3.1 *Definitions:*

3.1.1 Where reasonable, precise terms and names have been used within this guide. However, certain terms and names with varying definitions are ubiquitous within the literature and industry of vadose zone monitoring. For purposes of recognition, these terms and names have been included in the guide with their most common usage. In these instances, the common definitions have been included in Appendix X1. Examples of such terms are soil, lysimeter, vacuum and pore-liquid tension.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 Appendix X1 is a compilation of those terms used in this guide. More comprehensive compilations, that were used as sources for Appendix X1, are (in decreasing order of their usage):

3.2.1.1 Terminology D 653,

3.2.1.2 *Compilation of ASTM Terminology,*[3]

3.2.1.3 *Glossary of Soil Science Terms*, Soil Science Society of America,[4] and,

3.2.1.4 *Webster's New Collegiate Dictionary,*[5]

4. Summary of Guide

4.1 Pores in the vadose zone can be saturated or unsaturated. Some samplers are designed to extract liquids from unsaturated pores; others are designed to obtain samples from saturated pores (for example, perched ground water) or saturated macropores (for example, fissures, cracks, and burrows). This guide addresses these categories. The sampler types discussed are:

4.1.1 Suction samplers (unsaturated sampling), (see Section 7),

4.1.2 Free drainage samplers (saturated sampling), (see Section 8),

[1] This guide is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.21 on Ground Water Vadose Zone Investigations.

Current edition approved April 15, 1992. Published June 1992.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Compilation of ASTM Terminology*, Sixth edition, ASTM, 1916 Race Street, Philadelphia, PA 19103, 1986.

[4] *Glossary of Soil Science Terms*, Soil Science Society of America, 1987.

[5] *Webster's New Collegiate Dictionary*, Fifth edition, 1977.

4.1.3 Perched ground water samplers (saturated sampling), (see Section 9), and

4.1.4 Experimental absorption samplers (unsaturated sampling), (see Section 10).

4.2 Most samplers designed for sampling liquid from unsaturated pores may also be used to sample from saturated pores. This is useful in areas where the water table fluctuates, so that both saturated and unsaturated conditions occur at different times. However, samplers designed for sampling from saturated pores cannot be used in unsaturated conditions. This is because the liquid in unsaturated pores is held at less than atmospheric pressures (see *Richard's outflow principle*, in Appendix X1).

4.3 The discussion of each sampler is divided into specific topics that include:

4.3.1 Operating principles,

4.3.2 Description,

4.3.3 Installation,

4.3.4 Operation, and

4.3.5 Limitations.

5. Significance and Use

5.1 Sampling from the vadose zone may be an important component of some ground water monitoring strategies. It can provide information regarding contaminant transport and attenuation in the vadose zone. This information can be used for mitigating potential problems prior to degradation of a ground water resource **(1)**.[6]

5.2 The choice of appropriate sampling devices for a particular location is dependent on various criteria. Specific guidelines for designing vadose zone monitoring programs have been discussed by Morrison **(1)**, Wilson **(2)**, Wilson **(3)**, Everett **(4)**, Wilson **(5)**, Everett et al **(6)**, Wilson **(7)**, Everett et al **(8)**, Everett et al **(9)**, Robbins et al **(10)**, Merry and Palmer **(11)**, U.S. EPA **(12)**, Ball **(13)**, and Wilson **(14)**. In general, it is prudent to combine various unsaturated and free drainage samplers into a program, so that the different flow regimes may be monitored.

5.3 This guide does not attempt to present details of installation and use of the equipment discussed. However, an effort has been made to present those references in which the specific techniques may be found.

6. Criteria for Selecting Pore-Liquid Samplers

6.1 Decisions on the types of samplers to use in a monitoring program should be based on consideration of a variety of criteria that include the following:

6.1.1 Required sampling depths,

6.1.2 Required sample volumes,

6.1.3 Soil characteristics,

6.1.4 Chemistry and biology of the liquids to be sampled,

6.1.5 Moisture flow regimes,

6.1.6 Required durability of the samplers,

6.1.7 Required reliability of the samplers,

6.1.8 Climate,

6.1.9 Installation requirements of the samplers,

6.1.10 Operational requirements of the samplers,

6.1.11 Commercial availability, and

6.1.12 Costs.

6.2 Some of these criteria are discussed in this guide. However, the ability to balance many of these factors against one another can only be obtained through field experience.

7. Suction Samplers

7.1 Table 1 presents the various types of suction samplers. The range of operating depths is the major criterion by which suction samplers are differentiated. Accordingly, the categories of suction samplers are as follows:

[6] The boldface numbers in parentheses refer to the list of references at the end of this standard.

TABLE 1 Suction Sampler Summary

Sampler Type	Porous Section Material	Maximum[A] Pore Size (μm)	Air Entry Value (cbar)	Operational Suction Range (cbar)	Maximum Operation Depth (m)
Vacuum lysimeters	Ceramic	1.2 to 3.0 (1)[A]	>100	<60 to 80	<7.5
	PTFE	15 to 30 (2)[A]	10 to 21	<10 to 21	<7.5
	Stainless steel	NA[B]	49 to 5	49 to 5	<7.5
Pressure-vacuum lysimeters	Ceramic	1.2 to 3.0 (1)[A]	>100	<60 to 80	<15
	PTFE	15 to 30 (2)[A]	10 to 21	<10 to 21	<15
High pressure-vacuum lysimeters	Ceramic	1.2 to 3.0 (1)[A]	>100	<60 to 80	<91
	PTFE	15 to 30 (2)[A]	10 to 21	<10 to 21	<91
Filter tip samplers	Polyethylene	NA[B]	NA[B]	NA[B]	None
	Ceramic	2 to 3 (1)	>100	<60 to 80	<7.5
	Stainless steel	NA[B]	NA[B]	NA[B]	none
Cellulose-acetate hollow-fiber samplers	Cellulose Acetate Non cellulosic	<2.8	>100	<60 to 80	<7.5
	Polymer	<2.8	>100	<60 to 80	<7.5
Membrane filter samplers	Cellulose Acetate	<2.8	>100	<60 to 80	<7.5
	PTFE	2 to 5	NA[B]	NA[B]	<7.5
Vacuum plate samplers	Alundum	NA[B]	NA[B]	NA[B]	<7.5
	Ceramic	1.2 to 3.0	>100	60 to 80	<7.5
	Fritted glass	4 to 5.5	NA[B]	NA[B]	<7.5
	Stainless steel	NA[B]	49 to 5	49 to 5	<7.5

[A] Pore size determined by bubbling pressure (1) or mercury intrusion (2).
[B] NA = Not available.

7.1.1 *Vacuum Lysimeters*—These samplers are theoretically operational at depths less than about 7.5 m. The practical operational depth is 6 m under ideal conditions.

7.1.2 *Pressure-Vacuum Lysimeters*—These samplers are operational at depths less than about 15 m.

7.1.3 *High Pressure-Vacuum Lysimeters*— (Also known as pressure-vacuum lysimeters with transfer vessels.) These samplers are normally operational down to about 46 m, although installations as deep as 91 m have been reported **(15)**.

7.1.4 *Suction Lysimeters With Low Bubbling Pressures (Samplers With PTFE Porous Sections)*—These samplers are available in numerous designs that can be used to maximum depths varying from about 7.5 to 46 m.

NOTE 1—The samplers of 7.1.1, 7.1.2, 7.1.3, and 7.1.4 are referred to collectively as suction lysimeters. Within this standard, lysimeter is defined as a device used to collect percolating water for analyses **(16)**.

7.1.5 *Filter Tip Samplers*—These samplers theoretically have no maximum sampling depth.

7.1.6 *Experimental Suction Samplers*— The samplers have limited field applications at the present time. They include cellulose-acetate hollow-fiber samplers, membrane filter samplers, and vacuum plate samplers. They are generally limited to depths less than about 7.5 m.

7.2 *Operating Principles*:

7.2.1 *General*:

7.2.1.1 Suction lysimeters consist of a hollow, porous section attached to a sample vessel or a body tube. Samples are obtained by applying suction to the sampler and collecting pore-liquid in the body tube. Samples are retrieved by a variety of methods.

7.2.1.2 Unsaturated portions of the vadose zone consist of interconnecting soil particles, interconnecting air spaces, and interconnecting liquid films. Liquid films in the soil provide hydraulic contact between the saturated porous section of the sampler and the soil (see Fig. 1). When suction greater than the soil pore-liquid tension is applied to the sampler, a pressure potential gradient towards the sampler is created. If the meniscuses of the liquid in the porous segment are able to withstand the applied suction (depending on the maximum pore sizes and hydrophobicity/hydrophilicity), liquid moves into the sampler. The ability of the meniscuses to withstand a suction decreases with increasing pore size and also with increasing hydrophobicity of the porous segment (see 7.6). If the maximum pore sizes are too large and hydrophobicity too great, the meniscuses are not able to withstand the applied suction. As a result, they break down, hydraulic contact is lost, and only air enters the sampler. As described in 7.6, ceramic porous segments are hydrophilic and the maximum pore sizes are small enough to allow meniscuses to withstand the entire range of sampling suctions. Presently available polytetrafluoroethylene (PTFE) porous segments are hydrophobic, the maximum pore sizes are larger, and only a very limited range of sampling suction can be applied before meniscuses break down and sampling ends (see 7.6.1.3). Therefore, samplers made with PTFE porous segments may be used only for sampling soils with low pore-liquid tensions **(12, 17)**.

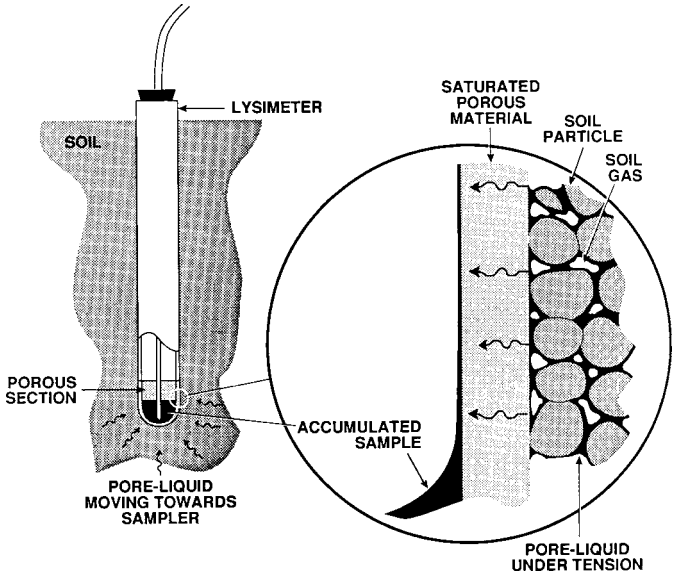

FIG. 1 Porous Section/Soil Interactions

7.2.1.3 The ability of a sampler to withstand applied suctions can be directly measured by its bubbling pressure. The bubbling pressure is measured by saturating the porous segment, immersing it in water, and pressurizing the inside of the porous segment with air. The pressure at which air starts bubbling through the porous segment into the surrounding water is the bubbling pressure. The magnitude of the bubbling pressure is equal to the magnitude of the maximum suction that can be applied to the sampler before air entry occurs (air entry value). Because the bubbling pressure is a direct measure of how a sampler will perform, it is more useful than measurement of pore size distributions.

7.2.1.4 As soil pore-liquid tensions increase (low pore-liquid contents), pressure gradients towards the sampler decrease. Also, the soil hydraulic conductivity decreases exponentially. These result in lower flow rates into the sampler. At pore-liquid tensions above about 60 (for coarse grained soils) to 80 cbar (for fine grained soils), the flow rates are effectively zero and samples cannot be collected.

7.2.2 *Suction Lysimeters*:

7.2.2.1 Vacuum lysimeters directly transfer samples to the surface via a suction line. Because the maximum suction lift of water is about 7.5 m, these samplers cannot be operated below this depth. In reality, suction lifts of 6 m should be considered a practical maximum depth.

7.2.2.2 Samples may be retrieved using the same technique as for vacuum lysimeters or, for deeper applications, the sample is retrieved by pressurizing the sampler with one line; this pushes the sample up to the surface in a second line.

7.2.2.3 High pressure-vacuum lysimeters operate in the same manner as pressure-vacuum lysimeters. However, they include an inbuilt check transfer vessel or a chamber between the sampler and the surface. This prevents sample loss through the porous section during pressurization, and prevents possible cup damage due to overpressurization.

7.2.2.4 Suction lysimeters with low bubbling pressures are available in each of the three previous designs. The only difference between these samplers and the three previous designs is that these porous sections are made with PTFE. The

low bubbling pressure (and hence large pore size or hydrophobicity, or both) of PTFE constrains these samplers to soils that are nearly saturated (see 7.2.1.2 and 7.6.1.3).

7.2.3 *Filter Tip Samplers*—Samples are collected from a filter tip sampler by lowering an evacuated sample vial down an access tube to a permanently emplaced porous tip. The vial is connected to the porous tip and sample flows through the porous section and into the vial. Once full, the vial is retrieved.

7.2.4 *Experimental Suction Samplers*— Experimental suction samplers generally operate on the same principle as vacuum lysimeters with different combinations of porous materials to enhance hydraulic contact. The samplers are generally fragile and difficult to install. As with vacuum lysimeters, they are generally limited to depths of less than about 7.5 m.

7.3 *Description*:

7.3.1 *Vacuum Lysimeters*:

7.3.1.1 Vacuum lysimeters generally consist of a porous cup mounted on the end of a tube, similar to a tensiometer. The cup is attached to the tube with adhesives (**18**[7]) or with "V" shaped flush threading sealed with an "O" ring. A stopper is inserted into the upper end of the body tube and fastened in the same manner as the porous cup or, in the case of rubber stoppers, inserted tightly **(12)**. To recover samples, a suction line is inserted through the stopper to the base of the sampler. The suction line extends to the surface and connects to a sample bottle and suction source in series. Body tubes up to 1.8 m long have been reported **(15)** (see Fig. 2).

7.3.1.2 Harris and Hansen **(19)** described a vacuum lysimeter with a 6 mm by 65 mm ceramic porous cup designed for intensive sampling in small areas.

7.3.1.3 A variety of materials have been used for the porous segment including nylon mesh **(20)**, fritted glass **(21)**, sintered glass **(22)**, Alundum (manufacturer name), stainless steel (**23**[7]), and ceramics (1.2 to 3.0 µm max pore size) (**18**[7]). The sampler body tube has been made with PVC, ABS, acrylic, stainless steel **(24)** and PTFE (**18**[7], **25**[7]). Ceramic porous segments are attached with epoxy adhesives or with flush threading. The stopper is typically made of rubber **(12)**, neoprene, or PTFE. The outlet lines are commonly PTFE, rubber, polyethylene, polypropylene, vinyl, nylon, and historically, copper. Fittings and valves are available in brass or stainless steel.

7.3.2 *Pressure-Vacuum Lysimeters*:

7.3.2.1 These samplers were developed by Parizek and Lane **(26)** for sampling deep moving pollutants in the vadose zone. The porous segment is usually a porous cup at the bottom of a body tube. The porous cup is attached with epoxy adhesives (**18**[7]) or with "V" shaped flush threading sealed with an "O" ring (**25**[7]). Two lines are forced through a two-hole stopper sealed into the upper end of the body tube. The discharge line extends to the base of the sampler and the pressure-vacuum line terminates a short distance below the stopper. At the surface, the discharge line connects to a sample bottle and the pressure-vacuum line connects to a pressure-vacuum pump. Designs are available that do not use a stopper but rather an

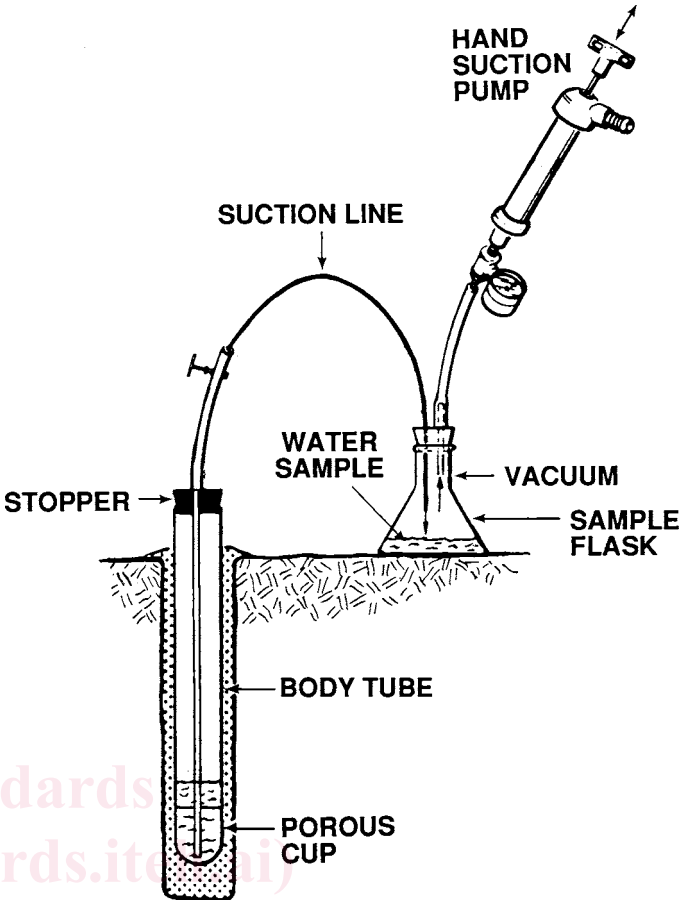

FIG. 2 Vacuum Lysimeter

"O" ring sealed, flush threaded top plug (**25**[7]). Tubing lines to the surface are attached to the top plug with threaded tubing fittings of appropriate materials. Body tubes are commonly available with 2.2 and 4.8 cm diameters and in a variety of lengths (see Fig. 3). The sampler and its components have been made out of the same materials used for vacuum lysimeters.

7.3.2.2 These samplers can retrieve samples from depths below 7.5 m because pressure is used for retrieval. However, during pressurization some of the sample is forced back out of the cup. At depths over about 15 m, the volume of sample lost in this fashion may be significant. In addition, at depths over about 15 m, pressures required to bring the sample to the surface may be high enough to damage the cup or to reduce its hydraulic contact with the soil **(27, 28)**. Rapid pressurization causes similar problems. Morrison and Tsai **(29)** developed a tube lysimeter with the porous section located midway up the body tube instead of at the bottom (see Fig. 4). This design mitigates the problem of sample being forced back through the cup. However, it does not prevent problems with porous segment damage due to overpressurization or rapid pressurization. The sleeve lysimeter (that is no longer available) was a modification to this design for use with a monitoring well **(1)** (see Fig. 5). Another modification is the casing lysimeter that consists of several tube lysimeters threaded into one unit (see Fig. 6). This arrangement allows precise spacing between units **(30)**.

[7] This reference is manufacturer's literature, and it has not been subjected to technical review.