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Automated System for Collecting Multiple, Sequential Samples from Soil Water Percolation Samplers under Continuous Vacuum

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Abstract: Manually collecting a series of sequential, discrete water samples from soil water percolation samplers, or similar devices that withdraw water from unsaturated porous media under continuous vacuum, is a logistical challenge, though the resulting collection can provide valuable information on the dynamics present in both laboratory and field studies. This article describes a sequential tension autosampler (STAS) that executes such sampling automatically. The STAS operates on 12 volts direct current (VDC) and can be adapted for laboratory and field applications. A data logger was programmed to operate a series of solenoid valves, which direct soil water collected under tension to seven individual collection bottles. The number of sequential samples, sample period, start time, and between-sample interval are specified by the user. The operator only need to attend the system periodically to transfer water samples to storage vials and program the next sampling sequence. In a laboratory study, the apparatus successfully collected samples overnight or over several days.

Keywords: Automation, lysimeters, percolation, sample fractionator, seepage, soil water, tension lysimeter, vacuum extraction

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INTRODUCTION

Vacuum-assisted soil water percolation samplers or tension lysimeters measure downward flux of water and solutes percolating through field soils. Continuous tension may be applied to percolation samplers at a constant potential via hanging water columns (Holder et al. 1991; Boll, Steenhuis, and Selker 1992; Zhu, Fox, and Toth 2002) or by pneumatic vacuum (Cole, Gessel, and Held 1961; Cochran, Marion, and Leaf 1970; Kincaid et al. 1979; Haines, Waide, and Todd 1982; Hergert 1986; Montgomery, Prunty, and Bauder 1987; Klocke et al. 1993). Continuous tension applied to samplers may also be varied to match ambient soil conditions at the sampler–soil interface, either manually (Brye et al. 1999) or automatically (Duke and Haise 1973; van Grinsven et al. 1988; Lentz, Sojka, and Kincaid 1998; Lentz and Kincaid 2003; Masarik et al. 2004). Instead of adjusting sampler extraction pressure, Kosugi and Katsuyama (2004) used a pulsing technique, switching sampler suction on when soil water tension in the native soil exceeded that in the sampler and off when the two tensions were equal. Research examining unsaturated flow processes commonly employ soil columns from which water is extracted under constant tension (Powelson, Simpson, and Gerba 1990; Phillips, Quisenberry, and Zeleznik 1995; Magesan et al. 1995; Vogeler et al. 1997).

The field and laboratory studies noted previously all require manual intervention to collect individual percolation samples. Thus, labor requirements and costs are increased when numerous samples are collected or when sampling during evening, early morning, or weekend hours. An automated sampling system would collect individual soil water samplers over time. This would allow researchers to better examine the dynamic character of soil water chemistry and transport, which may result from temporal changes in 1) soil temperature in response to diurnal forcing; 2) soil water percolation and solute concentration during rain storm events; or 3) the proportion of matrix-pore vs. macro-pore flow contributions to seepage.

Johnson et al. (2003) developed an automated sampling system for collecting soil water from zero-tension lysimeters, where water dripping from soil was captured in different individual containers over time. Chendorain and Ghodrati (1999) designed an automated instrument that performed continuous, real-time analysis of water extracted by peristaltic pump from a nearly saturated soil column ($\Psi_w > -2$ kPa). However, few, if any, automated, discrete sampling systems for continuous, controlled-tension extraction devices have been described in the literature.

The objective of this study was to develop a sampling system that automatically collected individual water volumes at specified times and intervals from unsaturated soil ($\Psi_w < -10$ kPa) while extracting the water under continuous pneumatic tension. The system's performance in a laboratory study is discussed.

MATERIALS AND METHODS

The STAS equipment was designed to sample water being withdrawn from an unsaturated porous media under continuous pneumatic suction. Thus, the STAS may be employed with fixed-tension, controlled-tension, or controlled suction-period lysimeters to monitor soil water dynamics. The STAS cannot be used with soil water samplers that apply tension via a hanging water column.

The STAS design employed a three-way solenoid valve, eight-way flow connector, and a series of valve controllers with two-way solenoid and Minimatic¹ valves (Figure 1, Table 1). This system received water collected under continuous tension from a soil water sampler and directed the flow *either* into a main sample collection vessel or into smaller collection bottles to collect a series of individual water samples. A programmable data logger controlled operation of the valves.

The three-way solenoid valve was mounted in the upper left corner of a clear acrylic sheet, which was mounted vertically on a wooden base (Figure 1). The eight-way flow connector (C1) was attached in the center of the acrylic backboard 5 cm below the top edge. A row of seven two-way, Teflon[®] solenoid valves (FV2) with 7.5-cm spacing was positioned on the acrylic backboard 10 cm below the eight-way flow connector. Holes drilled vertically into the backboard's wooden base and aligned with the two-way solenoid valves above held individual 100-mL Wheaton glass bottles. Teflon tubing (T1) conducted sample water between STAS components. Flexible plastic tubes (T2) connected each sample bottle to the principle vacuum source through a dedicated electric Minimatic valve and valve controller unit.

The data logger program operated each pair of Teflon solenoid (FV2) and Minimatic (FV1) flow valves via a digital output port, which activated a simple switching circuit in the valve controller (Figure 2). The same valve controller, sans the Minimatic valve, operated the three-way solenoid valve (FV3, Figure 1).

In the default state, water from the sampler flowed into the common port of the three-way solenoid valve (FV3) and out to the main collection vessel. When a sample was to be collected, the data logger activated the three-way valve, diverting source water flow away from the main collection vessel and into the eight-way connector. The program then simultaneously activated the dedicated pair of two-way solenoid and Minimatic valves. This applied a vacuum to the appropriate sample bottle and pulled a water sample into

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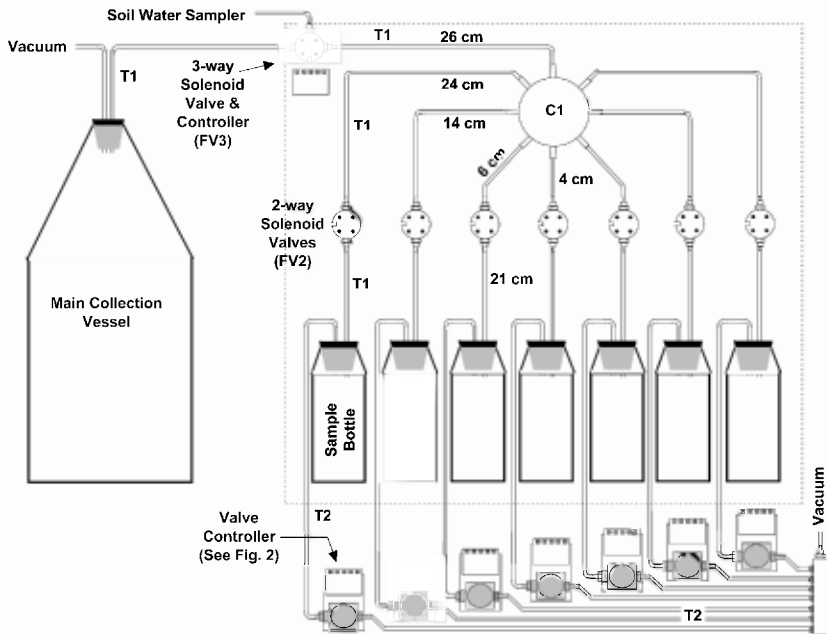


Figure 1. Diagram of sequential tension autosampler (STAS). The +DL and -DL labels refer to the 12-V terminals of the data logger.

the bottle by way of the now-open two-way solenoid valve and eight-way connector.

The water volume collected in sample bottles will vary with the collection period and flow rate through the soil water sampler. In field installations, the size of the sample bottles used and collection period selected must be matched to the field sampler flow rates to prevent overfilling of bottles during sampling.

Data Logger and Program

A program developed for a Campbell Scientific, Inc. (Logan, Utah) CR10X data logger executed the sample collection sequence. The program and associated documentation was described by Lentz (2005) and the data logger formatted file, Vaccntl3c.dld, can be downloaded at <http://www.nwisr1.ars.usda.gov/lentz/vaccntl3c.dld>.

The eight digital output, 12 VDC, and ground terminals on the CR10X wiring panel were used to operate the sampler. Digital output channels 1 to 7 controlled the solenoid and Minimatic flow valve sets for sample bottles 1 to 7, and output channel 8 controlled the three-way solenoid valve.

Table 1. Components list (partial) for sequential automated tension sampler, including valve controllers (manufacturer and part numbers in parentheses)

Label	Part	Quantity	Specification ^a
	Acrylic sheet mounting board	1	51 cm × 51 cm × 6 mm sheet
FV2	2-way direct-lift, teflon PTFE solenoid valve	7	12 VDC, 1.5 w, 1/4-28 ports, 0.088 Mpa vacuum rating (Cole-Parmer A-01367-70)
FV3	3-way direct-lift teflon PTFE solenoid valve	1	12 VDC, 1.5 w, 1/4-28 ports, 0.088 Mpa vacuum rating (Cole-Parmer A-01367-72)
C1	8-way multiport, teflon PTFE connector	1	(Cole-Parmer C-06473-05)
C2	8-outlet manifold	1	1/8" NPT outlet, 1/4" NPT inlet (Cole-Parmer, A-31522-18)
T1	Teflon sample tubing		3.2 mm (1/8") OD, 1.6 mm (1/16") ID
T2	Nalgene tubing		0.4 mm (1/4") OD, 3.2 mm (1/8") ID
Valve controller parts			
FV1	Minimatic electronic air-flow valve	7	Normally closed, 2-way, 12 VDC, 0.67 w (Clippard Minimatic ET-2M12-H[B])
M1	Flow valve manifold	7	Single supply w/1/8" NPT inlet, 10-32 port outlet (Clippard Minimatic 15490-2)
K1	SPDT relay	8	12-V, 5-amp (NTE R46-503-12)
Q1	Transistor	8	2N2222A (NTE123AP)
D1	Diode	8	1N4148
R1	Resistor	8	1.5 k ohm, ± 5%

^aVDC = volts direct current; NPT = National Pipe Thread; OD = outside diameter; ID = inside diameter.

The number of available output channels (eight) on the CR10X limited the maximum number of discrete samples taken per sequence to seven. Sample capacity of the instrument could be increased by adding extra sample-bottle/controller units, increasing the number of data logger digital output control ports using an expansion module, and modifying the data logger program to accommodate a greater sample number. To initiate a sampling sequence, the user inputs the day of year, hour (0-23), and min (0-59) the sampling sequence is to begin; the number of samples to be collected (1-7); interval between sampling (1-2879 min), length of sample period (1-2879 min); and day of year, hour (0-23), and min (0-59) sampling is to cease. The program outputs most of these parameters to the data logger's final storage area, in addition to the actual start and stop times for each

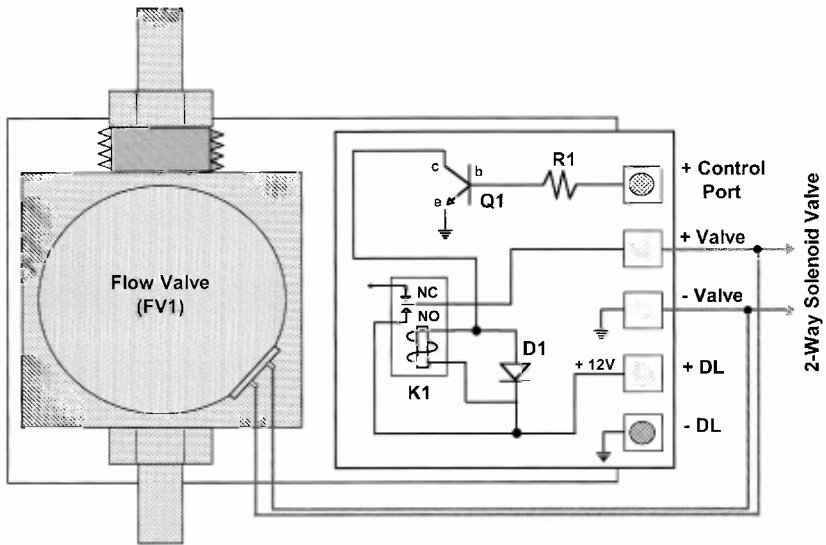


Figure 2. Schematic of the valve controller and minimatic air-flow valve.

sample collected. The sampling program must be reset at the start of each new year.

Testing STAS Operation

The system was tested in the laboratory while drawing water samples under continuous tension from a walled soil water percolation sampler (Lentz and Kincaid 2003). The percolation sampler consisted of a 23-cm-deep, 20-cm-diam. stainless steel beaker. Water was extracted via a tube through a 17-cm-long, 4-cm-diam. ceramic cup, which was embedded in a 5-cm-deep layer of silica flour in the bottom of the steel beaker. In field installations, the lip of the beaker is inserted into the roof of a cavity dug into the side of a soil pit. Thus, a continuous column of soil extends from the soil surface downward and into the steel beaker, where it contacts a soil layer placed over the silica flour bed.

In this laboratory test of the STAS, the walled percolation sampler was set up on a laboratory benchtop. A 12-mm-deep soil layer was placed over the silica flour bed in the steel beaker. A syringe pump delivered an aqueous solution to the soil surface in the beaker at a constant rate of nearly 1 mL/min, and water was withdrawn from the sampler's ceramic cup under 14 to 16 kPa tension. One to three sample collections were run for various programmed sampling periods, ranging from 3 to 24 h. Because water was drawn

from the sampler at a constant rate, the accuracy of the autosampling sequence was evaluated by comparing sample masses.

RESULTS AND DISCUSSION

The STAS successfully collected samples at the times, and over the periods, programmed by the user. Standard deviations of the sample masses for a given sampling series ranged from 0.6 to 1.5 g, representing coefficient of variation values from 0.6 to 1.7% (Table 2). These slight variations among samples were caused by small fluctuations in the suction applied to the sampler and not to timing differences during collection. Suction applied to the sampler was adjusted to 16 kPa at the start of each minute using a control system adapted from Lentz and Kincaid (2003). After the adjustment was completed, suction in the system was allowed to fall in response to incoming water and a slight amount of air leakage that occurred at fittings and connections. System suction typically did not fall below 14 kPa before the next adjustment cycle restored it to the preset value. Some variation in the pattern of falling suction was expected during sampling periods and likely produced the observed variation in sampled water volumes. System components are rated for 200 kPa pressures; hence, the STAS system should function equally well at the higher suction levels commonly used in field-installed soil water samplers.

The dead volume in the tubing and eight-way connector downstream from the three-way valve stored 0.75 mL of water from the previous sampling, which contaminated the subsequent sample. Assuming a 90-mL sample volume and 10-fold difference in solute concentration between contiguous samples, this contamination could alter the true solute concentration by -0.3% or $+7\%$, depending whether solute concentration increased or decreased between samples. If this error is excessive for one's application, its magnitude could be reduced significantly by placing the three-way solenoid valve immediately upstream from the eight-way connector.

Table 2. Average mass and standard deviations of water samples collected for programmed sampling sequences having various sampling intervals and periods

Sampling interval (min)	Sampling period (min)	Number of series evaluated	Mean sample mass (g)	Mean std. deviation of samples in similar series (g)
180	100	3	98.3	1.2
240	90	3	89.2	1.5
360	90	2	88.8	0.7
720	90	3	89.1	1.2
1440	90	1	88.3	0.6

When the STAS was reset for the next sampling sequence, any fluid that remained in the tubing between the three-way valve and sample bottles was purged. The tubing at the valve was detached, sample bottles disconnected, and the Teflon solenoid valves opened, allowing fluid to drain out of the system. One must not forget to reconnect the tubing to the three-way valve and reattach bottles after purging, else no subsequent samples can be collected. To eliminate problems associated with dead-space volume, a second three-way valve may be inserted after the first (in line with the eight-way connector). This valve would allow air into the system for a few seconds at the end of the sampling period, permitting any sample liquid remaining in the dead-space volume to drain down into the sample bottle. One of the data logger's digital output ports would be required to control the additional valve, reducing the number of available sampling bottles from seven to six.

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