

## A COMPARISON OF CATION SAMPLING IN FOREST SOILS

### BY TENSION AND TENSION-FREE LYSIMETERS<sup>1/</sup>

James H. Miller<sup>2/</sup>

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**Abstract.**--Field tests conducted in two soils with ceramic cup, ceramic plate, and tension-free lysimeters showed no concentration differences in collected cations (Ca, Mg, K, Na) between cups and plates, except for the hydrogen ion. Mean pH was 0.6 lower in cup collected samples for a sandy loam profile. Tension-free lysimeters of the design tested had persistent contamination from a glasswool component and yielded samples at a 60 cm depth from a clay loam profile and not from a loamy sand soil. Laboratory trails showed a marked response delay with plate lysimeters when sampling changing cation concentrations.

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### INTRODUCTION

Tension and tension-free lysimeters are being used more often to monitor natural processes in soil profiles and nutrient-loss impacts after cultural treatments. These devices permit solution sampling within and beneath undisturbed soil profiles. This cannot be done yet by other means. Tension lysimeters with porous ceramic cups have been used to monitor environmental contaminants (Parizek and Lane 1970, Nutter et al. 1978, Neary et al. 1979) and nutrient leaching processes (Vitousek et al. 1979, McColl 1978, Gosz and Dyck 1979). Tension plate techniques developed by Cole (1968) have clarified leaching mechanisms (McColl and Cole 1968) and nutrient losses following forest practices (Gessel and Cole 1965, Grier and Cole 1971). Jordon (1968) refined techniques using tension-free lysimeters and employed these to investigate nutrient transfers in tropical forest ecosystems (Kline and Jordon 1968; Stark and Jordon 1978). The investigative potential of such sampling devices is great, but the validity of cation sampling by the different lysimeters has not been fully explored.

The objective of this study was to compare tension-cup, tension-plate, and tension-free

lysimeters for quantifying nutrient losses from forest lands after vegetation management treatments. Each device was tested in the laboratory for response variation while sampling known concentrations of cations. A field study tested differences in concentrations from devices placed in isolated profiles with vegetation uptake eliminated. Sampling bias with the ceramic cup has already been shown for phosphate (Hansen and Harris 1975) and ammonium (Wagner 1962) which were not studied further here.

### MATERIALS AND METHODS

Ceramic cup and plate samplers used in this investigation are the commercial models manufactured by Soil Moisture Equipment Corp., Santa Barbara, California.<sup>3/</sup> The ceramic cup has a 2-bar bubbling pressure (or air entry value) with an outside diameter of 49.5 mm, an exposed length of 61 mm and ceramic thickness of 2 to 3 mm. Specified lengths of polyvinyl chloride (PVC) pipe are cemented to the cup permitting greater sampling depth and differing ratios of sample volume to vacuum-reservoir. The 60 cm length of PVC was tested here.

Plates are constructed of 0.5 bar ceramic with variable diameters of 265 to 275 mm and a thickness of 9 mm. A rubber disk with outport is attached to one side of the ceramic plate by peripheral wire and cement with a screen

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<sup>2/</sup>The author is a Research Forester with the Southern Forest Experiment Station, USDA--Forest Service, at the George W. Andrews Forestry Sciences Laboratory in Auburn, Alabama.

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interlayer to permit water flow. Connection between outport and a combination vacuum-sample bottle is by vacuum hose and copper tubing. Solutions collected with the plate must travel through 3 to 4 times more ceramic material than with the cup samplers, which have absorptive properties.

Ceramic cups are made from a non-vitreous porcelain which contains more or less equal proportions of kaolin, alumina ( $\text{Al}_2\text{O}_3$ ), ball clay, and other feldspathic materials.<sup>3/</sup> The  $\text{Al}_2\text{O}_3$  is essentially pure and 30% of the kaolin and ball clay are composed of this compound. Plates are 90%  $\text{Al}_2\text{O}_3$ , 6% kaolin and 4% ball clay. Kaolin and ball clay in both lysimeters are over 50% by weight  $\text{SiO}_2$  and have potential contaminants of iron, calcium (Ca), magnesium (Mg), potassium (K), sodium (Na), and titanium in amounts less than 1.5% each. Initial treatments of ceramic samplers are used to reduce the leachable or exchangeable cation contaminants (Gover and Lamborn 1970, Wolff 1967). I washed all ceramic samplers first with distilled water (DW) to remove ceramic dust, then 300 ml of 1N HCl was drawn through each, and finally, 1.2 liters of DW was tensioned through each lysimeter. This volume of DW was required before cation contamination reached zero.

Tension-free lysimeters were constructed of a 285 mm diameter funnel made by cutting off the upper portion of a 20-liter plastic carboy at the shoulder and inverting it on the bottom as a base. A drilled hole in the carboy cap with an epoxied connector served as an outport permitting tubing to be attached to a sample bottle. Glasswool filled the funnel and facilitated water flow (Stark and Jordon 1978). Repeated acid treatment (2% HCl) and DW washes of laboratory-grade glasswool was unsuccessful in eliminating cation contaminants. Average contaminant concentrations in a 300 ml DW wash were Ca 0.9 mg/l, Mg 0.5, K 0.2, and Na 2.0. Polyester fiber was also substituted for the glasswool and yielded similar contaminant concentrations. Due to this problem, tension-free data will be presented separately in the laboratory and field results.

In the laboratory trails, 300 ml aliquots of a premixed solution of four cations were sequentially drawn (0.4 bar tension) or passed (tension-free) through the samplers and analyzed using standard atomic absorption techniques. The solution concentrations were Ca 0.85 mg/l, Mg 0.45, K 0.23 and Na 3.10. Finally a 300 ml DW was then drawn or passed through each and analyzed. This procedure was repeated on three devices of each type to test the responsive capabilities of the samplers.

<sup>3/</sup> Personal communications with Mr. P. E. Skaling, Soil Moisture Equipment Corp.

In field comparisons, three lysimeters of each type were installed in a randomized complete-block design with three blocks in a loamy sand and three in a clay loam soil. The two installations are about 150 m apart and both are located in natural clearings under 53-year-old plantations of loblolly (*Pinus taeda*) and shortleaf pine (*P. echinata*). An access trench 1.5 m wide, 3 m long, and 1.5 m deep in the clearing centers was provided with a wooden shelter, with open sides for profile access. A sloping roof drained away from the installations. Tension plates and tension-free samplers were installed laterally in the trench walls. Cups were installed by auger holes using a 200-mesh silica sand interfacing material at the bottom of the hole. Lysimeters were emplaced at a 60 cm depth on the loamy sand site and at 45 cm on the clay loam site, below lateral rooting of the pines and equidistant from the access trench. A 30 cm wide and 1.5 m deep root exclusion trench surrounded the installation at a distance of 1.5 m from the access trench. Polythene sheeting was backfilled to the inside walls of the trenches to prevent root encroachment. All vegetation within each installation was carefully cleared and kept clear for the duration of the study. Thus plant uptake was eliminated as a variable.

A vacuum of 0.3 bars was maintained on tension devices over a collection period only during times of adequate soil moisture. At low soil moisture, cups maintained tensions more consistently than the plates, which required reevacuation periodically. Although cups maintained vacuum at lower soil moisture than plates, very little sample was collected during these periods. Tension-free lysimeters collected samples only after intense storm events.

Soil solution was collected 3 days after storm events or biweekly during extended rainy periods. Analyses included specific conductance Ca, Mg, K, Na, and pH. Specific conductance was measured with a conductivity meter with samples placed in a 25°C bath. Cations were determined by atomic absorption spectrophotometry. The pH was measured potentiometrically.

## RESULTS AND DISCUSSION

It is commonly thought that ceramic samplers reach equilibrium with the soil solution after installation. Thus, the process of adsorption, diffusion, and screening which biased phosphate sampling (Hansen and Harris 1975) merely delay the actual changes in soil solution concentrations when monitoring a profile. This detection delay can readily be seen in the laboratory results with the plate samplers (Fig. 1). With Ca, 1.5 liters of a 0.85 mg/l solution were drawn through the plates before the premixed sample concentrations were approached. Other cations reached equilibrium within 600 ml.

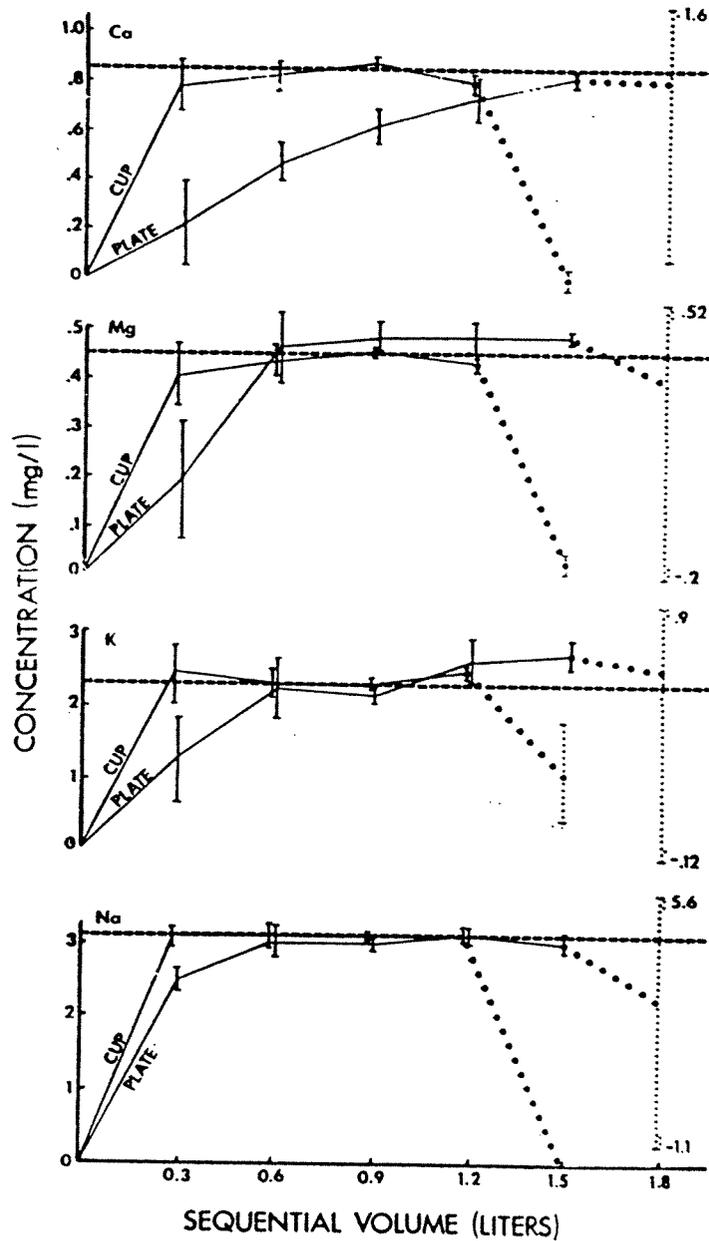


Figure 1. Mean concentrations of samples drawn from a known solution (concentrations indicated by horizontal dashed lines) and the sample standard deviations (vertical lines). The dotted lines indicate a final distilled water wash (0.3 liters) and the subsequent wash concentration.

Concentrations higher than the test solutions were found in samples for Mg and K. This phenomenon and the variable concentrations in the DW, evident by the large standard deviations, must result from desorption of bound ions. The large standard deviation also shows this desorption response to vary greatly from plate to plate. The plate with the slowest concentration response had the slowest average flow rate.

No percolating water reached the tension-free sample containers at the sandy site.

Using sample concentrations from the 12 storms as observations, a two-factor analysis of variance tested differences (1%) between soils and between cup and plate lysimeters. An additional analysis of variance and Duncan's multiple range tests were performed on the three lysimeter types using the tension-free collections in the clay loam soil.

Table 1. Mean cation concentration in solution washes of known concentration and a final wash of DW through tension-free lysimeters.

Solution	Ca	Mg	K	Na
	-----mg/l-----			
Premixed Concentration	0.85	0.45	0.23	3.1
1	7.52	1.05	.72	15.8
2	3.44	1.83	.67	15.1
3	2.21	1.09	.38	7.4
4	1.60	.80	.26	4.8
5	1.42	.71	.23	4.2
DW wash	2.82	1.32	.73	8.1

Cup lysimeters showed a more rapid response to drastic concentration changes. Although deviations from test resolution concentrations of  $\pm 1\%$  are apparent, the accuracy of analysis approximates  $\pm 1\%$  at these concentrations. Potassium desorption or leaching in the DW wash of cups points to a source of sampling bias with decreasing concentrations.

Persistent contamination was in the solution washes and DW washes passed through the tension-free devices (Table 1). Sodium was most concentrated. These devices had been previously washed with over 20 liters of DW in an attempt to eliminate the glasswool contaminants. The premixed concentration of K was finally reached after 1.5 liters, but absorbed or bound ions of all cations were leached in the DW wash.

Twelve storm periods were sampled at the field sites between July and November 1979. The longest storm was two weeks and the shortest 3 days. All devices had been previously installed and samples collected for at least 2 months before the July sampling commenced. This waiting period allowed for ample equilibration time. If equilibration of the ceramics does take place, then solution concentrations collected from both devices in the uniformized soil profiles should be the same. Collection from tension-free lysimeters were only made at the clay site during the sampling period.

Mean values for the 12 collections presented in Table 2, show no significant differences (1% level) between cups and plates for specific conductance, and Ca, Mg, K, and Na concentrations. The two samplers in loamy sand and clay loam soils, showed that total ionic concentrations were less in the clay soils as evident by significantly lower mean specific conductance. Significantly lower Ca concentrations appear responsible for the lower specific conductance in the clay profile. Two of the plate lysimeters in the sandy profile consistently gave higher concentrations for Mg and Na and correspondingly lower values for Ca and K. This appears to be endemic profile variation and was not found to be significantly different. Thus, monitoring of the most concentrated cations does not differ between lysimeter types at the concentration levels tested with an in vivo situation.

The only significant difference was pH. Plate lysimeters consistently collected samples that were .2 to .6 pH units higher. Due to the logarithmic nature of the pH scale, hydrogen ion activity was 1.5 to 4 times greater with the cup samplers. It would appear that selective absorption or screening or hydrogen ions was occurring with the plates and an equilibrium between the soil solution, ceramic plate, and sample solution had not been reached.

Sample contamination is evident in the mean concentrations from tension-free devices presented in Table 2. Magnesium concentrations were the only cations sampled that were not different from cup and plate samples. The inability to sample below rooting-zone depths in loamy sand soils plus the contamination problems with the glasswool polyester fiber will preclude the use of this sampler type in future forest nutrient-loss research.

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Table 2. Specific conductance, pH, and cation concentrations collected from two soils using three lysimeter types.

Soil	Lysimeter	Spec. Cond.	Ca	Mg	K	Na	pH
		$\mu\text{mhos/cm}$					
Loamy Sand	Cup	102a <sup>1/</sup>	5.8a	2.1a	0.9a	1.6a	5.8a
	Plate	117a	4.1a	3.4a	.4a	1.9a	6.4b
Clay Loam	Cup	60b	1.0b	2.0a	.9a	1.5a	5.9a
	Plate	65b	1.0b	1.9a	1.1a	1.4a	6.1b
	Tension-free <sup>2/</sup>	200c	4.1c	2.7a	2.3b	24.3b	7.5c

<sup>1/</sup>Means in a column followed by the same letter are not significantly different at the 1% level.

<sup>2/</sup>Tension-free data compared to the cup and plate collections in the clay loam soils only.

#### CONCLUSIONS

The cup lysimeter is the most reliable device for sampling cations, although a bias in phosphate and ammonium sampling has been demonstrated for these samplers (Hansen and Harris 1975; Wagner 1962). If the type of plate lysimeter studied in this investigation is used, then initial samples may not reflect actual soil solution concentrations due to plate adsorption. Plate-collected samples will not reflect accurate concentrations when drastic seasonal changes in soil solution concentrations occur. Cups are easier to install but plates have the distinct advantage of quantifying nutrient loss on an area basis due to the flat shape. The use of both cups and plates is needed to accurately sample cations and given an areal-loss estimate.

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