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Environmental Monitoring Branch
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Study #279: Characterizing solute movement in coarse-textured, leaching-vulnerable soils with zero-tension column lysimeters

I. Introduction

Lysimeters are devices that are typically incorporated into the soil to measure movement of water or solute. They are commonly used for agricultural purposes to account for evapotranspirative losses by determining the change in soil-water storage over time, usually by weight differential. Soil lysimeters also have been utilized to sample drainage water for solute concentration in contaminant transport research. In this context, soil water is collected either by gravitational means at zero tension into a reservoir at the bottom of the lysimeter or by an active process such as suction by capillary movement of water into a reservoir through a porous, ceramic-type interface with the soil. Zero tension soil lysimeters are widely utilized in parts of Europe for higher tier assessments of pesticide leaching potential to provide data for use in their pesticide registration processes (FOCUS, 2009). Suction lysimeters have been used by registrants and by the Department of Pesticide Regulation (DPR) to investigate aspects of pesticide movement in the soil, but zero tension lysimeters have not been widely utilized in US studies to characterize leaching of water and solute.

Historically, DPR field studies investigating movement of pesticides in the soil have focused on soil coring following pesticide and water applications. In these studies, chemical analysis of soil cores with respect to the depth at which they were collected has been used to characterize the fate and movement of pesticides in the soil. While these studies have provided valuable information contributing to the understanding of pesticide movement in the soil, soil coring alone has limitations in characterizing the fate and movement of leaching residues:

- 1) In irrigated agriculture, soil-water movement and leaching of residues are dynamic processes whereas soil coring is intermittent and potentially difficult to coincide exactly with leaching events.
- 2) Soil coring requires intensive use of staff resources and is often a limiting factor in the scale and scope of field study design.
- 3) Soil coring requires prior knowledge of potential residue movement in order to recover leaching residues at their maximum soil depth. The magnitude and intensity of water applications, pesticide physical/chemical properties and soil characteristics all influence movement of residues in the soil.

- 4) Analytical methods for soil-bound residues are typically less sensitive than for methods with residues in solution.
- 5) Pesticide transformation products can be more mobile than their parents presenting difficulties in accounting for their presence in soil cores when they are formed slowly and leached rapidly at very low concentrations.

Zero-tension, column lysimeters with collection reservoirs address these limitations inherent with soil coring:

- 1) All leachate within the confines of a column lysimeter is captured irrespective of the magnitude of water applications, potential for residue movement, or characteristics of the soil.
- 2) Sampling from lysimeters is relatively cost effective requiring less staff resources compared to soil coring; only requiring extraction of the solute by pump, with the frequency of sampling unrestricted.
- 3) Pesticide transformation products have been elusive in soil coring studies conducted by DPR, possibly due to their slow rate of formation coupled to limitations associated with their analytical reporting limits in soil and potential for movement. Lysimeters will intercept and accumulate leaching residues of transformation products.
- 4) When the base of the lysimeter soil core is below the soil evaporative depth, solutes captured in lysimeter reservoirs reflect a direct measurement of leaching residues. Such measurements are very applicable when evaluating the leaching potential of chemicals or the performance of models predicting leaching residues.

One requirement for utilizing zero-tension column lysimeters for characterizing leaching residues is that the soil core within each unit should be largely undisturbed and that the hydrological characteristics of each core are not significantly influenced by its contact with the lysimeter casing. Comparing solute concentrations from lysimeter reservoirs with residue concentrations sampled from unconfined soil cores is problematic leading to approaches of indirect comparisons (Kasteel et al., 2010). Subsequently, a more common approach to assess the leaching characteristics of soils within lysimeters has been to compare measured solute concentrations from their reservoirs to model-simulated solute concentrations from unconfined soils. FOCUS (2009) cited numerous studies investigating this subject with somewhat inconsistent results and opinions. A complicating aspect when comparing these studies was the diversity of soil types, lysimeter designs, study methodologies, modeling tools and solutes used.

Hardy et al. (2008) reported of comparisons between lysimeter studies and model leaching simulations for many pesticides that were assessed as part of a European regulatory decision making scheme to protect ground water. Many of these comparisons were in agreement (84%) and would have resulted in the same regulatory decisions being made. However, the comparisons were only qualitative in nature being judged as either exceeding or not exceeding a European ground water threshold concentration.

In some studies cited by FOCUS (2009) soil water content measurements and simulation results were compared between lysimeter-confined and unconfined cores, and where minor differences were discovered they were often attributed to a boundary-layer-effect at the base of the soil core within the lysimeter; this soil boundary condition being saturated due to its discontinuity in soil pore capillarity. Others have cited no appreciable difference in soil water content between lysimeter-confined and unconfined soil cores that were subjected to water inputs (Kasteel et al., 2010). Yet in this study, the transport of two pesticides with contrasting soil adsorption properties arrived at the lysimeter reservoirs (1.2 m deep) simultaneously and much earlier than simulations predicted, inferring preferential flow. Among other possibilities, the authors speculated that the lysimeter casing and possibly the saturated soil-boundary-effects at the base of the lysimeter-confined soil cores were influential. The soil used by Kasteel et al. (2010) was fine textured, boarding on a silty-clay- to silt-loam, and under field conditions could be expected to form cracks and fissures during expansion and contraction cycles. Furthermore, such fine-particulate soils would experience relatively strong capillary forces.

Efforts have been attempted at minimizing the potential effect of a saturated soil boundary layer at the base of a lysimeter-confined soil core in order to simulate a semi-infinite soil column (Corwin and LeMert, 1994). The authors were using a repacked, fine loam soil and a saturated lower boundary condition was likely of particular concern to them. Various grades of drainage material were installed between the base of the soil core and the solute-collecting reservoir, ranging from fine sand to gravel. A minor vacuum pressure could also be applied to the base of the lysimeter. As the effects of these modifications were not the objectives of their study, the impact of the drainage material and vacuum pressure was not reported.

As part of DPR's pesticide registration process, the Environmental Monitoring Branch (EM) evaluates the potential of pesticide active ingredients exceeding the specific numerical values (Johnson, 1989) to move to ground water based on their predicted behavior in the environment. The LEACHM pesticide fate and transport model (Hutson and Wagenet, 1992) coupled to an empirical-based model provides estimates of pesticide concentrations in well water resulting from simulated agricultural applications (Troiano and Clayton, 2009). The modeling scenario for these pesticide evaluations is representative of the large agricultural area in eastern Fresno and Tulare Counties where the ground water has been heavily impacted by pesticides. The soil in this area is coarse-textured and has been shown to be vulnerable to pesticide leaching under irrigated conditions (Troiano et al., 1993).

DPR's current pesticide modeling scenario has been verified against ground water monitoring data from the Fresno and Tulare County test area (Spurlock, 2000). In that study, however, model verification was over a large contiguous area encompassing 16 townships. The empirical component to DPR's pesticide modeling scenario simulates residue movement in the deep vadose zone and aquifer. This component is initialized with output from LEACHM, which simulates residue movement through the more complex surface soil layers containing organic matter, crop root systems and evapotranspirative processes. Lysimeters provide an opportunity to assess LEACHM performance directly by

comparing predicted leachate against measured leachate at the localized field scale and without the large influence of the modeling scenario's empirical component. This information would be useful for DPR field studies focusing on the effects of pesticide movement in the surface soil layers. EM is proposing such a study to investigate pesticide movement through turf in order to more thoroughly evaluate turf-based pesticides submitted for California registration. Also, future modifications proposed for the model's empirical component include utilizing input distributions for depth to ground water and residue aging in the aquifer. Currently, nominal input values are used for these two input parameters. Ideally, verification of predicted pesticide leaching in the surface soil layers by LEACHM would precede any evaluation of distributional inputs in the deeper vadose zone and aquifer on the overall model performance.

In summary, data from this study will determine whether lysimeters can be utilized successfully for future DPR field studies investigating the persistence and mobility of pesticides in the soil environment. These data also can be utilized to directly assess the performance of LEACHM and other pesticide fate and transport models to predict drainage water and leaching residues. Finally, the detection of degradation products in soil coring studies is often infrequent or at such low concentrations that concerns about their potential impact on ground water have been dismissed. Yet degradation products of several pesticides found in California ground water are detected more frequently and at higher concentrations than their parent compounds. We theorize that the relatively slow transformation process of parent to degradate would yield only very low levels of degradate in the soil at any one time, particularly if the degradate is mobile and escapes the maximum soil coring depth. Quantification of degradation products captured within lysimeter reservoirs may explain some aspects related to their presence in California ground water.

II. Study Objective

The objectives of this study are:

- 1) Determine if the soil confined within zero-tension column lysimeters is representative of the surrounding soil with respect to solute movement.
- 2) Evaluate the performance of the LEACHM model to predict drainage water and solute concentration for bromide and a number of pesticides under two diverse irrigation regimes.
- 3) Compare the movement and persistence of various degradates with respect to their parent pesticides.

III. Personnel

Study personnel from the Environmental Monitoring Branch of DPR include:

Project Leader:	Murray Clayton and Vaneet Aggarwal
Field Coordinator:	Alfredo DaSilva
Senior Scientist:	John Troiano

Project Supervisor: Lisa Ross
Laboratory Liaison: Sue Peoples for analyses conducted by CDFA
Cooperators:
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IV. Study Plan

This study will be conducted on a bare, coarse textured, sand, loamy sand or sandy loam soil. A contracted cooperator will be responsible for designating the location for the experiment, installation and testing of irrigation systems, application of chemicals via chemigation along with obtaining any necessary pesticide application permits and/or notifications to the County Agricultural Commissioner, maintenance, and irrigation of plots for the duration of the experiment. The contractor will assist with installation of the lysimeters. DPR staff will be responsible for the construction and installation of lysimeters, soil coring activities, solute sampling from lysimeters, chemical analysis, and data analysis.

The study will consist of two adjacent sites with treatment plots at each site arranged as a completely randomized design. Each site will contain eight, 1-m² treatment plots with four treatment plots randomly assigned a zero-tension column lysimeter to be installed at their centers and the remaining four treatment plots assigned as controls (Figure 1). Adjacent plots will be separated by 1 m. The irrigation system and lysimeters will be installed approximately four to six weeks prior to chemical application to the plots. The lysimeter design features and study soil properties will reflect characteristics that will minimize the potential for preferential flow and saturated lower boundary conditions. These lysimeter features will include the containment of undisturbed soil as opposed to repacked soil and a fine sand filtration barrier at the base of the soil core to improve drainage (Figure 2). The soil properties will be coarse textured, also to minimize the potential for preferential flow and saturated lower boundary conditions. After the irrigation system is verified for uniformity of water application, frequent irrigations will be conducted across the sites until drainage water is extracted from all lysimeters to confirm their functionality and standardize each plot's soil-water content. Simazine, atrazine, diuron, bromacil, norflurazon and hexazinone will then be applied by chemigation.

Chemical applications will be at maximum labeled rates. Potassium bromide will be applied at a rate of 100 kg Br ions/ha as a tracer for water movement. These chemicals will be applied simultaneously, or in the event of mixture incompatibility, sequentially and incorporated into the soil with a total of 1 inch of water. Irrigation will be applied to the sites at 7-day intervals for a period of approximately 60 days. One site will receive water applications at 100% of cumulative reference evapotranspiration (ET_o) to ensure that most of the bromide residues are maintained within the soil core. The other site will receive water applications at 160% of cumulative ET_o, which represents inefficient applications and likely typical of unpressurized surface delivery systems used in California (California Agricultural Technology Institute, 1988; Snyder et al., 1986). Inefficient water applications

to pesticide-treated sites are considered to be largely responsible for pesticide movement to ground water in leaching vulnerable areas of California. Irrigation will be indexed to ETo determined from a nearby CIMIS weather station. Collection of daily weather station data will include ETo; mean, maximum and minimum air temperature; and rainfall.

Sampling activities will consist of soil core sampling from lysimeter and control plots and solute sampling from lysimeter reservoirs (detailed methodology provided in Protocol Section V):

- *Sampling activities for objective #1 (comparison of bromide movement in soil cores between lysimeter and control plots):*

Approximately 60 days following bromide application and 7 days after the final water applications to the sites all lysimeter and control plots will be cored to collect soil samples. Soil cores from the lysimeter plots will be obtained from within the lysimeters themselves (Figure 1). These cores will be analyzed for bromide residues.

- *Sampling activities for objective #2 (evaluation of modeled versus measured water drainage and solute residues in lysimeter reservoirs):*

A single soil core will be collected near the center of each study site to confirm the absence of detectable background pesticide residues. These cores will be collected approximately three months prior to pesticide application. The specific location will be noted to ensure that it will not be overlaid by an experimental plot.

For modeling purposes the soil at each site will be characterized for various hydrogeological properties. Following irrigation and lysimeter installation six soil cores will be collected in three pairs at dispersed locations within each study site. Their locations will be centered between plots to ensure that the soil in each plot remains undisturbed. These soil samples will be used to characterize the soil's hydraulic conductivity, soil water retention parameters, textural composition, total organic carbon content, bulk density and initial soil moisture content. The samples will also be used to characterize background bromide levels.

Solute will be extracted from lysimeter reservoirs at 7-day intervals, occurring on the same day, but just prior to the weekly water applications. The water samples will be measured for total volume and analyzed for bromide and pesticide residues.

- *Sampling activities for objective #3 (comparing the fate of pesticide degradates with their parent products in soil cores and lysimeter solute):*

Data requirements to meet objective #3 will be achieved by sampling activities stated for objectives #1 (soil sampling for pesticide residues) and objective #2 (solute sampling from lysimeter reservoirs).

V. Sampling Methods

- Soil to be analyzed for background pesticide and bromide residues will be sampled using methods in soil sampling protocol FSSO002.00 (Garretson, 1999). These cores will be sampled to a depth of 3 feet at 6-inch increments. Upon extraction each 6-inch subsample to be analyzed for pesticide residues will be placed in a sealed jar on dried ice and maintained in frozen storage until chemical analysis. Samples to be analyzed for bromide residues will be sealed in plastic bags and transferred to refrigerated storage prior to analysis.
- Soil to be analyzed for pesticide and bromide residues from the lysimeter and control plots will be sampled following the general methodology in soil sampling protocol FSSO002.00 (Garretson, 1999). These cores will be sampled to a depth of 3 feet at 6-inch increments. Each 6-inch sub-core will be 12-inches in diameter (inside diameter of lysimeters) and extracted using post-hole diggers and trowels. Sanitizing of the soil extraction equipment will be consistent with those methods used for bucket augers as stated in sampling protocol FSSO002.00 (Garretson, 1999). Soil from each 6-inch sub-core will be thoroughly mixed inside a plastic bag and one of two subsamples of approximately 500 g transferred to a sealed jar on dry ice and maintained in frozen storage until chemical analysis. The remaining subsample will be transferred to a second plastic bag and sealed to be later placed in cold storage prior to its analyses for bromide residues using protocol [METH007.00](#) (Pinera-Pasquino, 2008).
- Soil to be analyzed for various hydrogeological properties will be sampled in three paired cores to a depth of 3 feet at 6-inch increments. One-of-each paired core will be collected using a sample ring kit designed to obtain undisturbed soil samples. Methods specified by the equipment manufacturer will be used to determine soil hydraulic conductivity (Eijkelkamp Agrisearch Equipment, Giesbeek, Netherlands), soil water retention, initial soil-moisture content and bulk density (Soil Moisture Equipment Corp., Santa Barbara, California, USA). The remaining paired cores will be sampled with standard bucket augers using soil sampling protocol FSSO002.00 (Garretson, 1999). These 6-inch sub-cores will be placed in plastic bags to be later analyzed for textural composition using protocol [METH004.00](#) (Dietrich, 2005), total organic carbon using protocol [METH005.00](#) (Gunasekara, 2006) and background bromide residues using protocol [METH007.00](#) (Pinera-Pasquino, 2008).
- Sampling from lysimeter reservoirs will consist of extracting all solute from each lysimeter using a manually operated, self-priming pump. Each extraction will be measured for total volume then partitioned into two vessels for pesticide and bromide analysis. The samples will be placed on ice then transferred to refrigerated storage until chemical analysis. Between each solute extraction the pump and its tubing will be flushed with cleansing liquids identical to those used for soil sampling equipment in protocol FSSO002.00 (Garretson, 1999).

VI. Chemical Analysis and Quality Control

Pesticide analysis will be conducted by the CDFA Center for Analytical Chemistry. A multi-analyte method is current for soil-bound and water solubilized simazine, atrazine, diuron, bromacil, norflurazon, hexazinone, the degradates of simazine and atrazine deethylsimazine (ACET), deisopropylatrazine (also ACET), didealkylated triazine (DACT), and the primary norflurazon degradate desmethyl norflurazon (DSMN). Analytical quality control procedures for these chemicals will follow recommendations from chemistry laboratory quality control protocol [QAQC001.00](#) (Segawa, 1995). Quality control procedures for the analysis of bromide in soil and water will follow those recommended in protocol [METH007.00](#) (Pinera-Pasquino, 2008).

VII. Data Analysis

Objective #1. This objective will test for a potential effect of the lysimeter on the distribution of bromide in the soil core.

Mixed Model Table

Source of Variation	Degrees of Freedom
Treatment (lysimeter vs control)	1
Soil Depth	5
Treatment x Soil Depth	5
Error	36

The statistical model used will be a repeated measures mixed model, where treatment and soil depth are fixed effects and soil core is a random variable. Soil core is a random variable because the results will be relevant for other test sites. The SAS model PROC MIXED allows for modeling of the variance component and it is anticipated that the variance model with respect to depth will require modeling because of autocorrelation in bromide concentration with depth. Various models will be explored with information criteria used to determine the best fitting model, such as AIC (Akaike Information Criterion). The assumption with this analysis is that significantly lower bromide residues in the lysimeter soil sub-cores would result from their preferential flow to deeper soil sub-cores or to the lysimeter reservoirs. Alternatively, similarity in bromide residue in corresponding sub-cores between lysimeter and control plots would indicate that preferential flow is not significant.

Objective #2. LEACHM-simulated leaching of water and bromide residues from which measured values from lysimeter reservoirs will be compared against will be of a deterministic type. The root mean square error (RMSE) will provide an assessment statistic. Simulated leaching of pesticide residues, also from which measured values from lysimeter reservoirs will be compared against will be probabilistic in nature as uncertainty in soil adsorption coefficients and field dissipation rates supports the use of distributional

inputs for these parameters. These distributions will be based on parameterized values given from Troiano (2009) and will be selected, where possible, from studies with soil types corresponding to the test site for this study. The RMSE based on the median value from the model output distribution and measured values from the lysimeter reservoirs will provide an assessment statistic.

A mass balance analysis will be performed for bromide using residues quantified from soil cores sampled within the lysimeters and solute extracted from their reservoirs.

For modeling purposes, soil water retention values derived from the analyses of the confined, undisturbed soil samples will be log-transformed and fit to soil hydraulic functions used in LEACHM by the SAS optimization procedure PROC OPTMODEL. Soil moisture content and bulk density will be determined from these samples for utilization in LEACHM input files. Their adjacent paired cores (unconfined samples) will be analyzed for textural composition and total organic carbon also for LEACHM parameterization.

Objective #3. A comparison of the mass of various degradation products recovered from the lysimeter reservoirs with their parent pesticides may provide some information on the relatively high frequency of detections and high concentrations of degradates found in California ground water. This study will provide a dataset on the leaching of transformation products needed for further model development.

VIII. Timetable of Activities

December 2011 / February 2012:

Finalization of study protocol.

March 2012 / April 2012:

Finalization of contract with cooperator.

April / May 2012 (April 15):

Day 1: Chemical analysis of soil cores for background pesticide and bromide residues.

Day 80-109 (July 4 – Aug 2):

Installation of irrigation system and verification of uniformity of water application.

Installation of lysimeters.

Conduct frequent irrigations over study plots (lysimeter and control plots) until all lysimeter reservoirs experience drainage to ensure their functionality and to standardize the soil-water content across all plots.

Day 110 (Aug 3):

Soil coring to characterize soil hydraulic properties, bulk density, textural composition, and total organic carbon content.

Day 111-175 (Aug 4 – Oct 7):
Laboratory analysis of soil samples to characterize soil hydraulic properties,
bulk density, textural composition, and total organic carbon content.

Day 111 (Aug 4):
Chemigation of pesticides and potassium bromide.

Day 112 (Aug 5):
First irrigation.

Day 119 (Aug 12):
First solute extraction from lysimeters.
Second irrigation.

Day 126 (Aug 19):
Second solute extraction from lysimeters.
Third irrigation.

Sequencing at 7-day intervals.

Day 168 (Sept 30):
Eighth solute extraction.
Ninth and final irrigation.

Day 175 (Oct 7):
Ninth and final solute extraction.
Soil sampling within study plots for chemical and bromide residues.

November / December 2012:
Chemical analysis.

January 2013 / April 2013:
Data analysis.

May 2013 / Aug 2013:
Reporting of study results.

IX. Budget

Budget component	Units	Expense/unit (\$)	Total expense (\$)
Contracted cooperator	1	40,000	40,000
Pesticide soil analysis of background residues	12	864	10,368
QA/QC for background residues	1	864	864
Pesticide soil analysis	96	864	82,944
QA/QC for pesticide soil analysis	10	864	8,640
Pesticide analysis of chemigation solute	3	864	2,592
QA/QC for pesticide analysis of chemigation solute	1	864	864
Pesticide analysis of lysimeter reservoir solute	72	864	62,208
QA/QC for pesticide analysis of lysimeter reservoir solute	7	864	6,048
Equipment & supplies (lysimeters)	8	500	4,000
Equipment & supplies (other)	1	2,000	2,000
Travel	1	2,000	2,000
PY	0.25	100,000	25,000
Total			247,528

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Figure 1. Randomized layout of control and lysimeter plots in each experimental site.

Site of water application
at 100% of ETo

Site of water application
at 160% of ETo

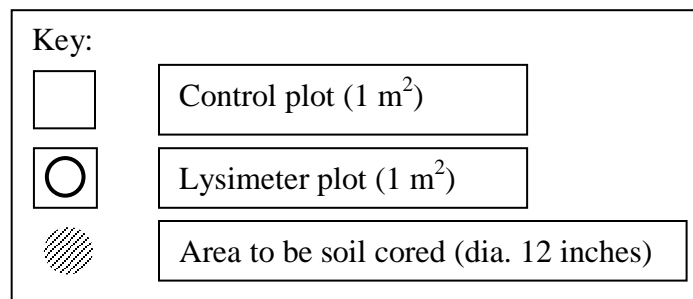
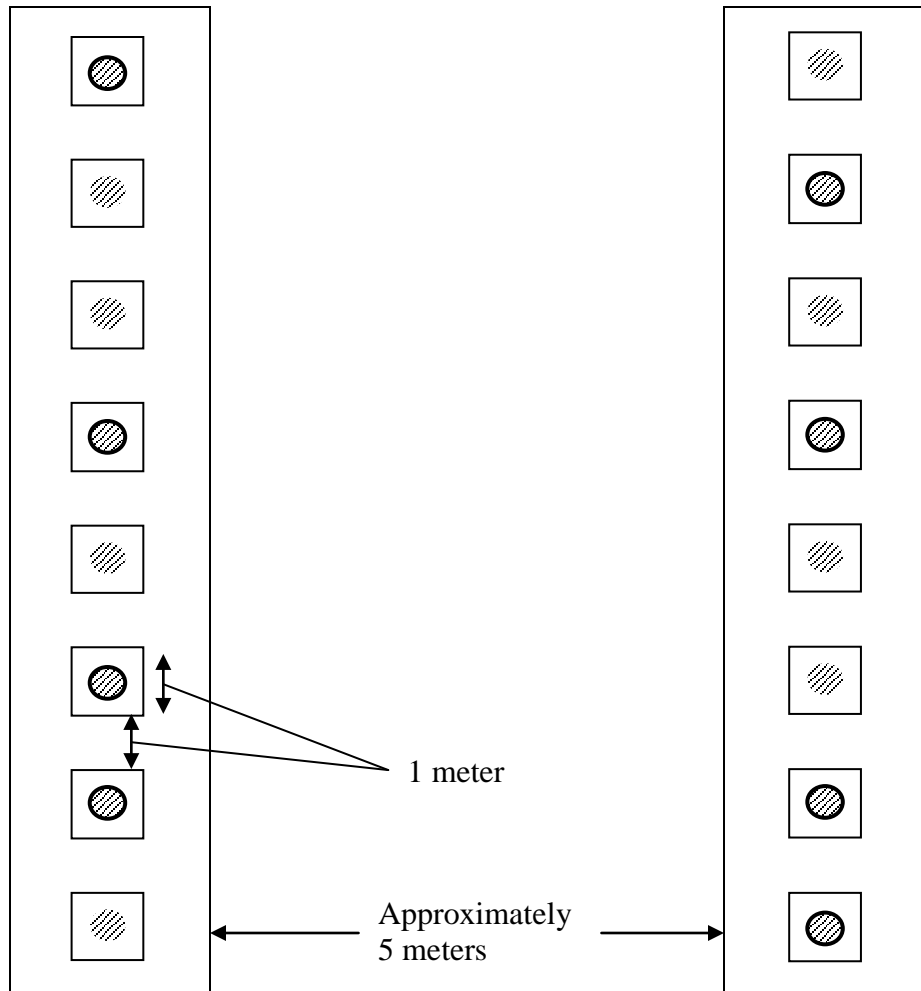


Figure 2. Lysimeter design.

