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**Guidance Document for the Performance
Of Out-door Monolith Lysimeter Studies**

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The work of the OECD related to chemical safety is carried out in the **Environmental Health and Safety Programme**. As part of its work on chemical testing, the OECD has issued several Council Decisions and Recommendations (the former legally binding on Member countries), as well as numerous Guidance Documents and technical reports. The best known of these publications, the **OECD Test Guidelines**, is a collection of methods used to assess the hazards of chemicals and of chemical preparations. These methods cover tests for physical and chemical properties, effects on human health and wildlife, and accumulation and degradation in the environment. The OECD Test Guidelines are recognised world-wide as the standard reference tool for chemical testing.

More information about the Environmental Health and Safety Programme and its publications (including the Test Guidelines) is available on the OECD's World Wide Web site (see page 8).

The Environmental Health and Safety Programme co-operates closely with other international organisations. This document was produced within the framework of the Inter-Organisation Programme for the Sound Management of Chemicals (IOMC).

The Inter-Organization Programme for the Sound Management of Chemicals (IOMC) was established in 1995 by UNEP, ILO, FAO, WHO, UNIDO and the OECD (the Participating Organisations), following recommendations made by the 1992 UN Conference on Environment and Development to strengthen co-operation and increase international co-ordination in the field of chemical safety. UNITAR joined the IOMC in 1997 to become the seventh Participating Organisation. The purpose of the IOMC is to promote co-ordination of the policies and activities pursued by the Participating Organisations, jointly or separately, to achieve the sound management of chemicals in relation to human health and the environment.

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FOREWORD

In 1993, the OECD Task Force on Ecotoxicology for priority setting in development and revision of Test Guidelines appropriate for pesticides testing gave priority to the development of a guidance document for out-door lysimeter studies. At their 5th Meeting in October 1994, the National Co-ordinators of the Test Guidelines Programme (TGP) endorsed the Task Force's recommendations and agreed to initiate the work in this area. At the 6th National Co-ordinators Meeting (NCM) in December 1995, Germany offered to take the lead in drafting the proposal.

In November 1996, a first draft proposal for an OECD Guidance Document on Performance of Out-door Lysimeter Studies, prepared by the BBA (Germany), was circulated to the National Co-ordinators and National Experts of the TGP for review. Eight countries (Austria, Canada, Germany, Japan, the Netherlands, Switzerland, the United-Kingdom and the United-States), industry (ECETOC, ECPA) and the European Co-operation Programme COST-66 made comments. The document was revised in light of comments received and submitted to National Co-ordinators of TGP for finalisation and approval at the 10th NCM in September 1998.

The revised proposal was well appreciated and the proposed methodology for lysimeter studies was generally fully accepted. However, several additional comments were made by Austria, Denmark, France, Japan, Switzerland, the Netherlands, the United-Kingdom, the United-States and ECETOC. The document was again revised taking into account the amendments suggested and then circulated to National Co-ordinators of TGP for final approval. In April 1999, the 11th NCM approved this last version and agreed that the draft Guidance Document be submitted to the Joint Meeting s for declassification.

The Joint Meeting of the Chemicals Committee and the Working Party on Chemical recommended that this document be made public. It is being published on the responsibility of the Secretary General of the OECD.

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INTRODUCTION

Man-made chemicals may reach soil directly via deliberate application (agrochemicals) or via indirect routes (e.g. via wastewater → sewage sludge → soil or air → wet/dry deposition). For risk assessment of these chemicals it is important to predict their potential for transformation in soil and for migration from the top soil layer to the subsoil and to groundwater.

The fate and behaviour of a chemical in soil can be estimated by laboratory tests on adsorption/desorption, soil column leaching, volatilisation, chemical degradation and microbial transformation. In certain cases (e.g. for mobile and/or persistent chemicals), it may be necessary to conduct experiments under outdoor conditions, for example in monolith lysimeters or field plots. Compared to laboratory tests, these higher Tier studies take into account more biological and physical factors along with their complex interactions.

Various types of lysimeters, differing in the filling technique and in the way the leachate is collected, are known, such as:

- monolith lysimeters versus artificially filled lysimeters (both with permeable bottom plate),
- zero-tension lysimeters (permeable bottom plate with simulated water table at that depth) or
- suction lysimeters (active collection of leachate through water-permeable material via suction pressure).

Monolith lysimeters (see definition in section: “*Lysimeter*”) have been used in research with crop protection products for some years. They are useful tools for obtaining information on the fate and behaviour of a chemical in an agricultural ecosystem.

This guidance is based on two national Guidelines (1)(2) and the outcome of international workshops held in Germany (3) and the USA (4).

DESCRIPTION OF THE TEST

Principle of the test

Monolith lysimeters are used to study the fate and behaviour of chemicals in an undisturbed soil profile under outdoor conditions. They allow for the monitoring of the volume of leaching/drainage water as well as the concentrations of a chemical and its transformation products therein. An outstanding feature of monolith lysimeters therefore is the capability to monitor mass fluxes of water and chemicals. Additionally, the distribution of the chemical and of its transformation products between the soil and plants along with their transformation rates can be determined.

Applicability of the test

Although most experience with monolith lysimeter testing has been gained with crop protection products, the test is applicable to all chemical substances (radiolabelled or unlabelled) for which an analytical method with suitable accuracy and sensitivity is available. Its applicability is not restricted by the physical properties of the test substance.

Any soil (agricultural or non-agricultural) with a deep profile, which allows the collection of an undisturbed monolith block can be used for the test. However, it may be difficult to collect undisturbed monoliths from heavy clays and stony soils with the technique described in the guidance document. Moreover, the shrinkage of clay soils can lead to cavities along the walls of the lysimeter and thus to rapid movement of water and chemicals directly to the bottom of the lysimeter (edge or wall effect). Therefore, such clay soils should not be used for lysimeter experiments.

This test method is not designed for application to paddy field situations.

Advantages and limitations of the test

Compared to laboratory experiments, out-door monolith lysimeter studies have the following advantages:

- they are closer to field environmental conditions, there is no significant disturbance of the subsurface soil (below the top 25-30 cm plough layer),
- it is possible to grow plants and therefore to study the fate of chemicals in soil/plant systems, transformation and leaching, which are normally measured separately in laboratory experiments, remain integrated processes,
- mass fluxes can be determined;

And the major limitation is:

- expense (depending on design): the fact that certain variable experimental conditions such as environmental/climatic parameters (temperature, rainfall, light and wind) are normally not controlled.

Compared to experiments in field plots, monolith lysimeters have the following advantages:

- the possible use of labelled chemicals (depending on specific country regulations);
- the achievement of a more complete material balance, as soil, plants and leachate can be analysed for test substance and transformation products (when labelled test substance is used);
- the possibility to detect lower amounts of the test chemical and its transformation products (when labelled test substance is used);
- some environmental factors (e.g. rainfall, solar radiation) could be controlled, if required.

And the limitations are:

- the bottom boundary between the soil block and the container influences the water flow¹ and thus can affect the amount of chemical leached from a lysimeter;
- the spatial variability is normally less (particularly when compared to field plots of $\geq 10\text{m}^2$);
- they are not suitable for every plant species;
- they are not suitable for every soil type.

Information on the test substance

Non-labelled or radiolabelled substances can be used for the studies. The use of ¹⁴C-labelled material is recommended for studies with crop protection products, as it will permit to establish a material balance and to detect and identify transformation products. As far as possible, the labelling position should be in the most stable part(s) of the molecule. The purity of the test substance should be at least 95 %.

The following information on the test substance should be available:

- water solubility [OECD Test Guideline 105];
- n-octanol/water partition coefficient [OECD Test Guideline 107 and/or 117];
- adsorption/desorption² [OECD Test Guideline 106] and/or leaching in soil² (OECD Test Guideline in preparation);
- dissociation constant in water [OECD Test Guideline 112];
- hydrolysis as a function of pH [OECD Test Guideline 111];
- vapour pressure [OECD Test Guideline 104] and Henry constant;
- aerobic transformation in soil² (draft OECD Guideline 307);
- photodegradation in soil.

An appropriate analytical method of known accuracy, precision and sensitivity for the quantification of the test substance and, if relevant, of its transformation products, must be available. The

1 The bottom layer of any lysimeter is on the average wetter than what can be actually observed in an undisturbed soil profile at a similar depth in the field. This disturbance of the water flow can be avoided by inserting suction cups with a permanent suction power (e.g. approx. 50 hPa) or a sand/gravel layer at the bottom of the lysimeter. But this drainage situation does not allow water from deeper subsoil layers to move upwards. For a more detailed discussion of these aspects see reference (5).

2 For interpretation of lysimeter results it would be useful to conduct studies on adsorption/desorption or soil column leaching and on aerobic transformation in the same soil as found in the top layer of the lysimeter.

limit of detection and of quantification for the test substance and its transformation products should also be known.

Description of the method

Lysimeter

A monolith lysimeter consists of an undisturbed soil block or cylinder, embedded in an inert container (e.g. stainless or galvanised steel, fibre glass) with a bottom permeable to drainage water or leachate (e.g. a perforated bottom, quartz sand filter bottom). A sampling device allows for collection of the leachate. Examples of monolith lysimeters used are shown in Figures 1 to 3.

The dimensions of the lysimeter, in particular the surface area, depend on the purpose of the study. In order to allow for the growth of plants, a minimum surface area of the soil core of 0.5 m² is recommended. To reduce edge effects and to enhance representativeness, a size of 1.0 m² is suggested. A larger surface area will also increase the number of plant species which can be grown in the lysimeter. A smaller surface area may be used for bare soil experiments

Also the depth of the soil monolith depends on the purpose of the study. For testing of crop protection products, the depth usually ranges from 1.0 to 1.3 m. Other core depths may be used depending on the distribution and thickness of the soil horizon of interest. The effects of a shallow water-table can be simulated by adjusting the height of the outflow (see Figure 2 in the Annex).

Collection of soil monoliths

Several techniques for collecting undisturbed soil monoliths are described in the literature. For details of these techniques consult references (6)(7)(8)(9)(10). The basic principles are described hereafter.

To collect an undisturbed soil monolith, the lysimeter casing (e.g. square, round, 8-edged, dodecagonal, etc.) is covered at the top with a steel plate, 30 mm thick. At the bottom, the walls of the casing (8 - 10 mm thick) have sharpened edges for cutting the soil. The casing is carefully pressed vertically into the soil to the desired depth by the shovel of an excavator as shown in Figure 4 (6). However, the wall of the casing should finally be 5 cm above the soil surface to avoid contamination of the surrounding areas by runoff of the test substance from the treated monolith surface during the study. With this technique it is possible to obtain soil monoliths without artificial cracks and gaps between the lysimeter walls and the soil (6).

After the casing has been pressed into the soil, the surrounding soil is removed and a carrying rack with a perforated bottom plate made of stainless or galvanised steel is placed besides the monolith. The bottom of the rack with the sharpened cutting edge is pressed underneath the casing (Figure 4). Then the lysimeter casing with the monolith standing in the rack is lifted from the hole and transported to the lysimeter station.

At the experimental station, the lysimeter casings are inserted into the containers permanently installed in the ground (Figure 5). A sand/gravel container (filled with fine sand and small gravel from top to bottom) may be used below the casing to ensure an undisturbed water flow out of the soil monolith (see Figure 3).

Soil

The type of soil selected depends on the purpose of the study. It should represent worst case conditions with regard to leaching, e.g. a light sandy soil with low adsorptive and water holding capacity allowing rather fast movement of chemicals and water through the soil profile. For crop protection products, agricultural soils related to use areas should be used. Non-agricultural soils may be used for the testing of general chemicals which may reach the soil via waste water-sewage sludge or air-wet/dry deposition.

For ionisable substances, the pH of the soil may have a significant influence on their behaviour in soil. Therefore, it is recommended that for acidic test substances with a $pK_a < 5.0$, the pH of the soil should be at least two pH units higher than pK_a .

The various horizons of the soil should be analysed for texture (percentage sand, percentage silt, percentage clay)³, pH, cation exchange capacity (CEC), organic carbon, water holding capacity and soil structure (e.g., aggregates, cracks, fissures, biopores, 'skeleton'). For determination of soil properties the methods recommended in references (12)(13)(14)(15)(16) can be used.

The soil used in the lysimeter should not have been treated with the test substance or a similar compound belonging to the same chemical class for a minimum of one year before monolith sampling to avoid analytical problems and/or adaptations of soil microflora.

Procedure

The lysimeter study is carried out under conditions representative of field conditions. This is achieved by embedding the container in a surrounding soil matrix which in fact is an experimental plot surrounding the lysimeter where all management measures (except application of radiolabelled test substance) are performed parallel to the lysimeter. After installation of the lysimeter, the soil monolith should rest for re-equilibration for sufficient time (at least three months, preferably one growing season) prior to application of the test substance. Monthly measurements of leachate volumes may indicate that replicate lysimeters behave consistently; this information can also be obtained from the use of tracers.

The experiments should be run at a location which is representative of the climatic conditions of the use area and favourable with respect to leaching. In order to attain representative microclimatic conditions on the lysimeter surface of planted lysimeters, guard crops should be grown in the surrounding area identical to that in the lysimeter (see Figure 3).

When using large lysimeters (surface area $\geq 0.5 \text{ m}^2$) at least two lysimeters (1) with the same soil should be run in parallel at the same time at the same location to detect possible anomalies between the replicates. In case of smaller surface areas ($< 0.5 \text{ m}^2$) a greater number of lysimeters may be necessary.

3 According to FAO and US soil classification system (11).

Application of the test substance

For crop protection products, formulations containing the radiolabelled test substance should be used. They should be applied according to their use recommendations as spray, as granule or as treated seed at the highest recommended rate of application in accordance with Good Agricultural Practice (GAP). For general chemicals, the application of the test substance should simulate their most relevant route of entry into soil (e.g. sewage sludge, wet/dry deposition).

For crop protection products, lysimeter studies should take into account the occurrence of multiple applications as follows:

- Multiple applications during one vegetation period (e.g. fungicides, insecticides): if multiple applications are recommended for one crop, or if its use in different crops within a crop rotation on the same field and in one vegetation period, can lead to multiple applications, the lysimeters should be treated in the same way regarding the application times and the highest recommended application rates. The suggested study duration is two years after the last application.
- Applications in two succeeding years: if applications are likely to occur in two succeeding years on the same field (e.g. herbicide on cereals or maize) two lysimeters should be treated with the highest recommended application rate. In the second year, one of the two lysimeters should be treated in the same way a second time and at the intended time. The suggested study duration for the two lysimeters is two years after the last application in the lysimeter treated twice.

Special care has to be taken when applying radiolabelled test substance to minimise losses and to avoid contamination of the surrounding areas in the lysimeter station. This can be done by surrounding the area to be treated, for example, by thin aluminium plates covered with a tin foil or by wire cages covered by plastic sheets. For further details see references (6)(7)(8)(9).

Tracer substances

For a better understanding of hydraulic conditions during the study it may be appropriate to use non-volatile tracer compounds which neither interact with the soil nor with the test substance. Mobile tracers include negatively charged ions or compounds such as chloride, bromide, iodide, the food-dye Brilliant Blue FCF or fluorescent dyes (e.g. Sulvoflavin). Sorbing tracers are mostly positively charged compounds (e.g. Methylene Blue, fluorescent dye Rhodamin WT).

Duration of the study

The duration of a lysimeter study should be determined by the objective of the study and the properties of the test substance but should normally be at least two years after the last application of the test substance (1). In some cases it may be appropriate to extend this period to three years, e.g. in case of annually repeated application of a crop protection product or for chemicals with half-lives greater than six months. The expected study duration could be derived from information gained e.g. from results on adsorption and degradation rates and from application pattern. It may also be appropriate to modify the duration according to the results obtained during the study.

Management

For crop protection products, the management (e.g. fertilisation, seed bed preparation, sowing, tillage and harvest) of the lysimeter including its surrounding area is carried out according to good agricultural practice (6) (8). Special attention has to be paid to the depth of soil tillage which should only be done in the top 25-30 cm (plough layer). In the case of testing general chemicals, management practices will depend on the purpose of the study.

Outdoor experiments are subject to natural climatic variations. Therefore, it may be necessary to complement natural precipitation⁴ by irrigation. Whenever this is needed, water with a quality comparable to rain water (e.g. rain, tap or well water) should be supplied to allow for plant growth. It is recommended that the pH and ionic strength of the irrigation water should be determined. Deionised water can destroy the soil structure and therefore must not be used. Daily portions provided should be at least 5 mm but not more than 20 mm. It is recommended to use artificial rainfall simulators to simulate natural rainfall and allow for slow infiltration. The irrigation rate should not exceed 0.5 mm • min⁻¹.

Environmental conditions

Precipitation should be recorded daily at the lysimeter site. Also soil temperature and soil moisture⁵ should be measured at 20 cm depth⁶; the measurements should be done in a separate lysimeter, in case the probes are installed vertically from the lysimeter surface.

Other weather data (air temperature, solar radiation, humidity, potential evaporation) should be obtained onsite or at a nearby meteorological station. Frequency and time of these measurements should be compatible with standard meteorological procedures (at least daily) as many estimation models for unknown parameters (e.g. evapotranspiration) rely on these standard data.

Sampling

Collection and sampling of leachate should preferably be done, if possible, on a weekly basis, unless excessive rainfall events allow shorter intervals. The sampling of leachate from the bottom of the lysimeter can be done through suction tubes (Figure 1), exhaust pipes (Figure 2) or by gravity drains (Figure 3). Small cores of top soil (0-10, 10-20, 20-30 cm) should be taken with an appropriate soil corer after each vegetation period. Number and diameter of soil cores depend on the surface area of the lysimeter and should not remove more than 1 % of the total surface area. At harvest time, plants including weeds grown in the lysimeter are also sampled.

At the end of the study the whole soil monolith should be horizontally dissected into layers of appropriate thickness (e.g. 25 cm). After intensive mixing of each soil layer, samples for analysis will be taken to identify the residue pattern over the whole soil profile. Alternatively, several soil cores can be taken from the soil monolith and segmented in layers for analysis.

Analysis

Leaching/drainage water should be analysed for the test substance and/or its transformation products, total radioactivity (when labelled test substance was used) and, if applicable, for tracer compound. If ¹⁴C-labelled test substance was used, leachate should also be analysed for ¹⁴C-CO₂ –

4 The precipitation varies with climatic region. For example, 800 mm is required as annual precipitation rate in Germany (1) which corresponds to an amount of leachate in of the order of $\geq 250 \text{ l/m}^2$.

5 Soil moisture can be determined, for example, by neutron probes and by time domain reflectometry. The use of tensiometers may be critical as they may dry in summer or the water in the ceramic cell may freeze in winter destroying the cell.

6 Measurements at two additional depths (e.g. 50 and 80 cm) may be helpful for the interpretation of lysimeter results.

carbonate and – bicarbonate which may have been formed upon mineralisation of the molecule or parts of it.

The various soil layers and plant material(s) are also analysed for the test substance and/or its transformation products and, when radiolabelled test substance was used, for total and non-extractable radioactivity.

Sample storage

If soil and/or leachate samples cannot be analysed immediately they should be stored deep-frozen in the dark. Conditions and length of storage should be reported. Also, the storage stability of the test substance and, if relevant, its transformation products should be determined.

Waste disposal

Excess samples of soil, drainage/leaching water and plants not needed for analysis but containing significant amounts of labelled material should be treated as waste according to country-specific regulations. This also applies to all extracts used for analysis and all extracted soil and plant samples.

Data and reporting

Test report

The report should give a full and comprehensive description of the study and its results. Along with a full description of the analytic techniques employed, the following data should be reported:

Information on test substance and relevant metabolites:

- purity of the test substance;
- specific radioactivity and labelling position(s) (if appropriate);
- method(s) for analysis of test substance and transformation products including limits of analytical detection/quantification.

Information on lysimeter:

- pedologic composition for all soil horizons including particle size distribution, organic carbon content, cation exchange capacity, pH -KCl;
- soil structure (e.g. aggregates, cracks, fissures, biopores, ‘skeleton’);
- size of surface area/depth of soil core;
- casing material;
- excavation technique for the monolith;
- installation of lysimeter;
- “history“ and location of the site from where the monolith was obtained (i.e. former treatment with crop protection products, fertilisers, other chemicals 3 years prior to collection);
- information on the soil moisture contents in the monolith prior to and at collection is desirable for a better interpretation of the test results;
- instrumentation for measurement of soil temperature and moisture;

Tracer(s) used, if any.

Application of test substance:

- time(s) of application;
- rate(s) of application;
- kind of formulation;
- application technique.

Maintenance activities:

- type of vegetation;
- agricultural practices (date of seeding, time of harvest, yields, etc.);
- treatment with other chemicals (amounts and dates of application of fertilisers, other crop protection products, etc.)

Conditions during the test⁷:

- geographical location;
- weekly mean of air temperature;
- weekly mean of soil temperature and soil moisture (measured at least at one depth of 20 cm and two additional depths, preferably, in a separate untreated lysimeter);
- monthly sum of precipitation/irrigation;
- irrigation technique (e.g. artificial rain, sprinkling, watering, etc.);
- annual sum of precipitation/irrigation;
- reporting of rainfall events during the first month after application on a daily basis;
- distribution of precipitation (snow, low-intensity rainfall, thunderstorms, etc.), if appropriate
- weekly potential evaporation (estimation).

Leachate:

- sampling technique including description of materials used;
- time of sampling and volume of all samples;
- annual total volumes.
- concentrations in leachate (in $\mu\text{g} \cdot \text{l}^{-1}$ and % of applied amount):
 - concentration of test substance in all samples;
 - concentration of transformation products in all samples;
 - concentration of unknown radioactivity in all samples, if appropriate;
 - concentration of tracer in all samples, if appropriate;
 - concentration of total radioactivity in all samples, if appropriate;
 - concentration of ^{14}C -labelled CO_2 /carbonate/bicarbonate in all samples, if appropriate;
 - total amounts of the test substance, transformation products, extractable residues and non-extractable radioactivity, if appropriate.

⁷ Full records of soil and weather data may be reported as an Appendix.

Residues in soil (in mg/kg dry weight and % of applied amount):

- concentration of test substance in soil layers;
- concentration of transformation products in soil layers;
- concentration of extractable radioactivity in soil layers, if appropriate;
- concentration of non-extractable radioactivity in soil layers, if appropriate;
- total amounts of the test substance, transformation products, extractable residues and non-extractable radioactivity, if appropriate.

Residues in plants (in mg/kg fresh weight and % of applied amount):

- concentration of test substance;
- concentration of transformation products;
- concentration of extractable radioactivity, if appropriate;
- concentration of non-extractable radioactivity, if appropriate;
- total radioactive residues, if appropriate.

Material balance (recovered percentage of applied dose or radioactivity at the end of the study).

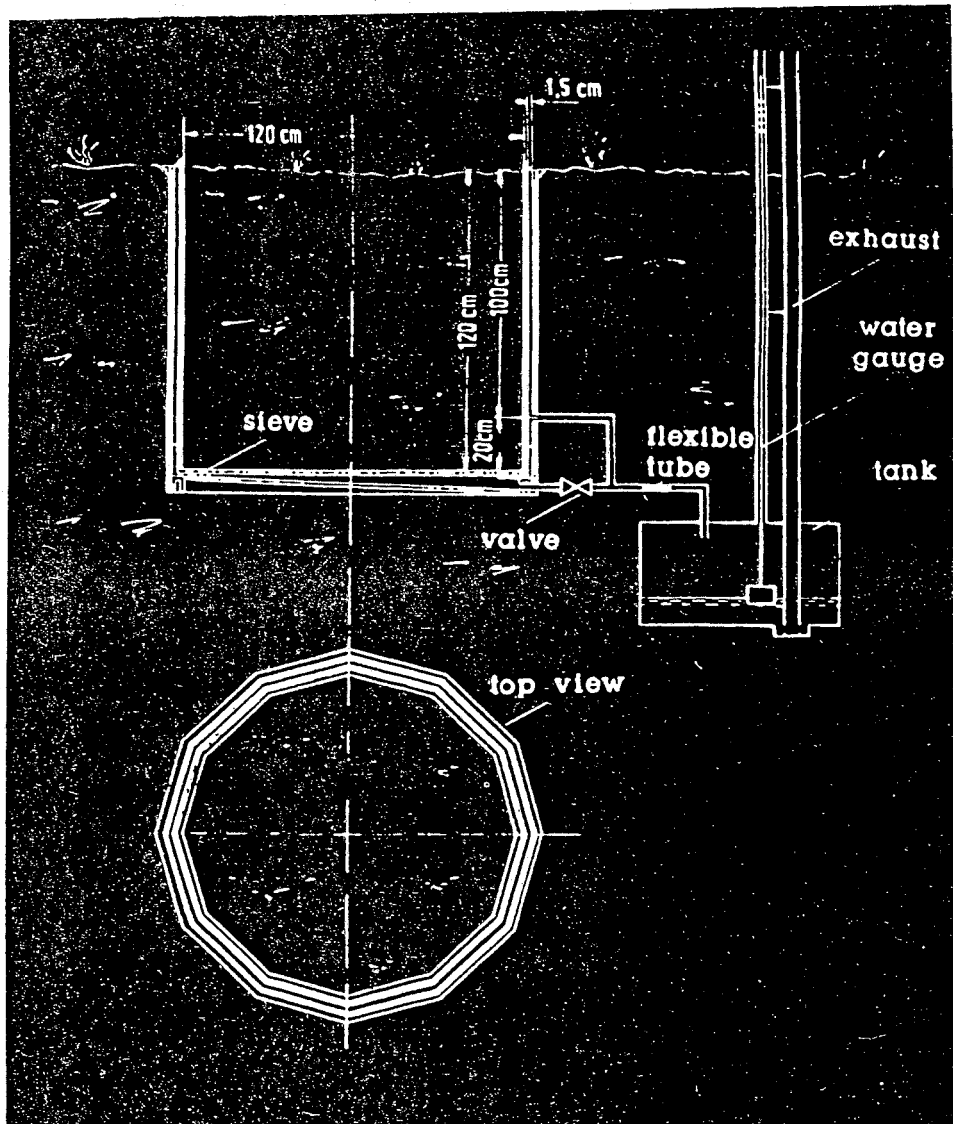
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Figure 2

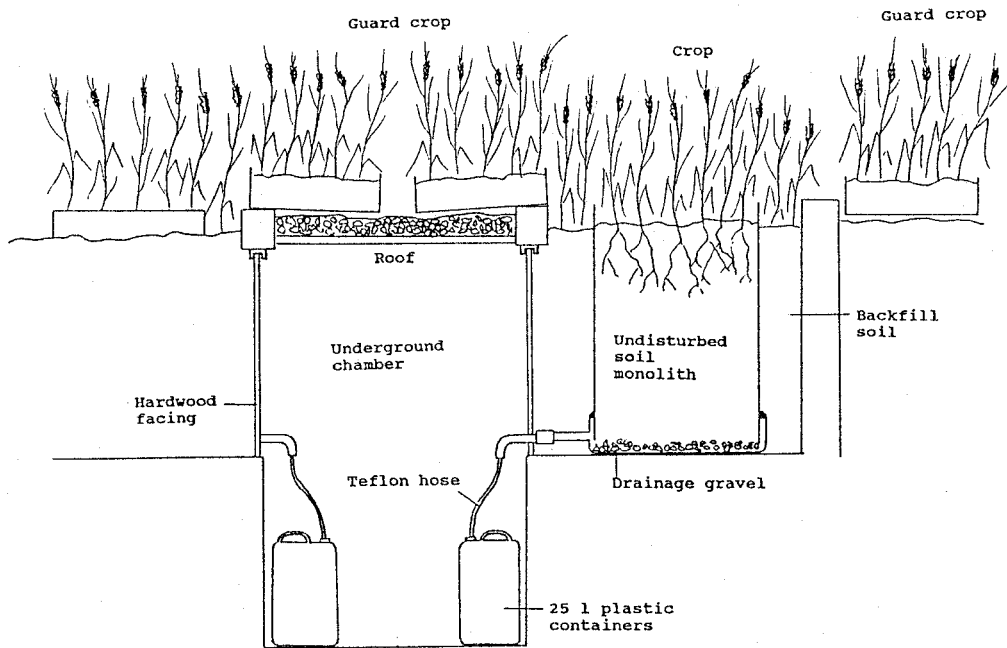
Example of a dodecagonal lysimeter system (7)



Example of a dodecagonal lysimeter system which allows to establish a groundwater level. The dodecagonal structure gives the lysimeter container a high stability. The lysimeter consists of an inner and an outer container of 1.0 to 1.5 m column length and 1 m² surface area. The outer container has two outlets for the leachate. A groundwater level can be simulated by closing the lower outlet. The leachate is collected in a separate tank connected to the container by a flexible tube and is sampled through the exhaust pipe. The lysimeters are embedded in the ground. To prevent animals (e.g. birds, mice) from entering the lysimeter field, the whole area is surrounded and covered by a wire-netting fence.

Figure 3

Example of a lysimeter with an underground compartment (9)



This is an example of a lysimeter installation with an underground compartment for collection of leachate or for creation of artificial water tables in individual lysimeters. Guard crops are grown in containers around the lysimeters to simulate the microclimate in field plots.*

* Leachate outlets at the bottom of the lysimeters should be located at the lowest point to allow free drainage of water. It may often be better to have a fine sand layer at the bottom instead of a gravel package because fine-sand barriers can be hydraulically connected to glass-fibre wicks (a water-saturated porous hanging water column that siphons excess water to the lower outlet end).

Figure 4

Filling of a lysimeter with an undisturbed soil monolith (6)



Figure 5

Insertion of a lysimeter casing into the container embedded in the ground of the lysimeter station (top) and total view of lysimeter station (below) (6)

