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Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
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# LONG-TERM BIOBARRIERS TO PLANT AND ANIMAL INTRUSIONS OF URANIUM TAILINGS

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## ABSTRACT

The objective of this project was to develop and evaluate the effectiveness of physical and chemical barriers designed to prevent plant and animal breachment of uranium mill tailings containment systems for an extended period of time.

A polymeric carrier/biocide delivery system was developed and tested in the laboratory, greenhouse and field. A continuous flow technique was established to determine the release rates of the biocides from the PCD systems; polymeric carrier specifications were established. Studies were conducted to determine effective biocide concentrations required to produce a phytotoxic response and the relative rates of phytotoxin degradation resulting from chemical and biological breakdown in soils. The final PCD system developed was a pelletized system containing 24% trifluralin, 18% carbon black and 58% polymer. Pellets were placed in the soil at the Grand Junction U-tailings site at one in. and two in. intervals. Data obtained in the field determined that the pellets released enough herbicide to the soil layer to stop root elongation past the barrier.

Physical barriers to subsurface movement of burrowing animals were investigated. Small crushed stone (1 to 1½ in. diameter) placed over asphalt emulsion and multilayer soil seals proved effective as barriers to a small mammal (ground squirrels) but were not of sufficient size to stop a larger animal (the prairie dog). No penetrations were made through the asphalt emulsion or the clay layer of the multilayer soil seals by either of the two mammals tested. A literature survey was prepared and published on the burrowing habits of the animals that may be found at U-tailings sites.

## EXECUTIVE SUMMARY

Twenty-three million metric tons of uranium tailings, containing 14,000 Ci of $^{226}Ra$, have been disposed of at several locations in the U.S. Stabilization of these tailings disposal sites is necessary to minimize human exposures to the products of $^{238}U$ decay. The tailings should also be protected from biotic intrusions such as plant roots and burrowing animals to prevent ground-water or food-web contamination.

This study was undertaken to develop and test physical and chemical barriers that would prevent animal and plant intrusion of radon seals placed over uranium mill tailings. Chemical barriers can be installed to prevent plant root intrusion into buried wastes, while physical barriers can provide protection against the actions of burrowing animals.

Barriers put into use would have to meet requirements of

- not damaging seals over the contained tailings, aboveground vegetation or other biota of the area.
- acting as an effective biobarrier to animal and plant intrusion for extended periods of time.

All development and testing of devices in this work treated these requirements as practical objectives.

Following the introduction to the report, our results are presented in six major sections. The first five deal with aspects of developing an effective chemical barrier for long-term use at a tailings disposal site. The final section deals with physical barriers to the intrusion of burrowing animals.

### TESTS OF CHEMICAL BARRIERS

Following development of a polymeric carrier delivery (PCD) device for the slow release of an herbicide (Section 2.0, Controlled Release Systems), we conducted feasibility tests of different herbicides and evaluated the effectiveness of these chemicals (Section 3.0). Results of these preliminary studies showed that roots in a PCD system treated with the herbicide trifluralin maintained their growth above the chemical barrier but did not penetrate the chemical barrier or the soil beneath it. For these reasons, trifluralin was chosen as the phytotoxin to be used in the final PCD system.

Studies of polymer systems for containing the herbicide (Section 4.0) followed our initial feasibility tests. Polymers in sheet and pellet form were used for these, and we discovered that the pellet form was most desirable. Pellets proved to be easily applied and didn't interfere with water movement in the soil, as did sheets. Moreover, a cylindrically shaped pellet (9 mm x 9 mm) could provide a sufficient trifluralin reservoir to maintain the chemical barrier for 100 years.

Because one of our objectives was to employ the minimum concentration of herbicide required to inhibit basipetal root growth, we conducted studies on the degree of toxicity of trifluralin as well as its rate of degradation (Section 5.0). We tested 13 species of native plants to determine a range of minimum effective levels for the herbicide and found these minimum levels to range from 0.3 to 6.4 ppm for the species tested. Tests of microbial and chemical degradation revealed that the natural half-life of trifluralin in soil was about 50 days. These tests also showed that trifluralin concentrations in the soil around the pellet reached equilibrium about 30 days after placement, and that a diffusion gradient of trifluralin in soil was established. Minimum effective levels of herbicide were maintained 4 cm to 6 cm from the device.

After laboratory tests were completed, we conducted field evaluations of PCD systems that had proved most promising in the laboratory (Section 6.0). Trifluralin was used in the pellet form; liquid treatments over asphalt seals were employed as well to establish the effectiveness of trifluralin as a deterrent to root intrusions of asphalt emulsion seals. Another objective here was to test the placement of trifluralin relative to the seals to determine the best barrier to root elongation under actual site conditions. We found that mixtures of trifluralin with asphalt negated the effectiveness of the herbicide in greenhouse tests (Section 2.0), though trifluralin sprayed on or above the seal prevented roots from reaching the surface of the seal. Liquid trifluralin (in the form of chemical's parent compound) remained effective for at least one year following placement beneath the soil surface. The pellet PCD system tested at Grand Junction, Colorado is performing well to date. Trifluralin remains in a thin layer above and below the PCD system.

## TESTS OF PHYSICAL BARRIERS

The final section of the report (Section 7.0) deals with physical barriers to the actions of burrowing animals. In this part of the study, we sought to identify species whose presence might pose a threat at established tailings sites and to test barriers for preventing intrusion by these animals. Barriers tested included a layer of asphalt covered with a layer of topsoil; successive layers of asphalt, crushed rock and topsoil; and a type of multilayer earth barrier consisting of layers of sand, bentonite clay, and rock. In these tests, we found that the ground squirrels, a small burrowing rodent, did not penetrate any of the barriers. Prairie dogs, however, were able to penetrate a rock barrier constructed from crushed stones 1 to 1½ in. in diameter.

## RECOMMENDATIONS

Most of the U-tailings sites, with one exception, are located in the western states and are characterized by arid climates with neutral to basic soils. The pelletized PCD system does not affect soil water movement; the trifluralin remains in a thin layer and is stable even under heavy irrigation. Because of this, we feel the PCD system can be applied successfully in most climatic regimes. We recommend placing the pellets in a uniformly spaced layer two or three feet below the soil surface.

We recommend the following procedures to be used in designing an animal intrusion barrier:

- identify the species of burrowing animals that live at the specific site
- determine their body weights and burrowing habits
- design a stone barrier accordingly.

Our studies show that a 6 in. layer of 1 to 1½ in. crushed stones placed 18 in. below the soil surface will stop ground squirrels having a body weight of 300 g or less. However, prairie dogs with average body weights of 2 kg can dig through crushed stone of this size. Actual size of stones to be used in a barrier for larger animals will have to be field tested.

If a properly designed asphalt emulsion seal at least 7 cm thick is used, plant and animal barriers are not needed for as long as the seal maintains its integrity. Both the PCD system as a root barrier and the stone layer as an animal barrier are recommended to use with multilayer earth seals, as plants and animals may damage the integrity of earth or clay seals.

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# 1.0 INTRODUCTION

## 1.1 PROGRAM HISTORY

Much of the uranium ore mined within the United States from the early 1940's through 1970 was processed for the Manhattan Engineering District (MED) and the Atomic Energy Commission (AEC) by private companies. When processing operations ceased and a mill became inactive, tons of uranium mill tailings remained. Mill operators were not aware of the potential radiation health hazards to the public from exposure to the mill tailings, and the general scientific concensus at the time was that the effects of the radioactivity on the public were minimal. However, radiological criteria guidelines became more stringent as research on the effects of low-level radiation progressed. As a result, an initial evaluation of inactive uranium mill tailings sites was begun in 1972 to determine radiological status and potential health effects that these sites might pose to the public. Since 1972, considerable effort has been directed towards recovering and regenerating radiological site status information and records, identifying sites and conducting engineering assessments, and developing legislation to initiate remedial action at inactive uranium mill tailings sites.

In 1972 Congress passed Public Law 92-314 (later amended by Public Law 95-236). This act provides authority and funds for a cooperative State/Federal Program to perform remedial actions on structures in Grand Junction, Colorado, where mill tailings were used for construction. A Department of Energy (DOE) report to Congress in February 1979, "Progress Report on the Grand Junction Mill Tailings Action Program" (DOE/EV-0033), provided an analysis of the current status of the program.

Also in March 1972, the Subcommittee on Raw Materials of the Joint Congressional Committee on Atomic Energy (JCAE), held hearings on bills that would have provided for a cooperative program between the Federal Government and State of Utah. The purpose of such a program would have been to implement remedial action in the area of the inactive uranium mill tailings sites, as opposed to treating the potential problem on a piecemeal basis. The outcome was an assessment of the existing physical conditions of inactive uranium mill sites located in eight western states by the AEC in 1974, in cooperation with the EPA and the affected states. Detailed engineering evaluations of many of these sites commenced in 1975 and were completed in 1977 by the Energy Research and Development Administration, the successor to the AEC.

In April 1978, DOE proposed legislation to Congress which would establish a program to stabilize and control the mill tailings in a safe and environmentally sound manner. Hearings on the proposed legislation began in June 1978 in conjunction with similar bills introduced in the Senate and House of Representatives.

As a result of these hearings, Public Law 95-604 was enacted on November 8, 1978. This act authorized DOE, along with the affected states, Indian tribes, and persons who owned or controlled inactive uranium mill tailings, to establish assessment and remedial action programs at inactive uranium mill tailings sites. Title I of that act further stipulates that DOE will meet all radiation standards promulgated by EPA. Additionally, DOE will finance 90 percent of the remedial action costs; the affected states will be required to pay the remaining costs from non-Federal funds. Indian tribal lands are an exception to this requirement; here, 100 percent of the costs for remedial action will be borne by the Federal Government.

Currently, greater than 14,000 Ci of $^{226}Ra$ are contained in about 23 million metric tons (MT) of tailings at 25 inactive uranium tailings sites in the western United States (U.S. Department of Energy 1979). The principal radiological implications and associated potential health hazards of these tailings appear to be related to the radionuclides of the $^{238}U$ decay chain, primarily $^{230}Th$, $^{226}Ra$, $^{222}Rn$ (a noble gas), and $^{222}Rn$ progeny. Decay products of $^{238}U$ with possible adverse effects to the general public from long-term exposure are $^{226}Ra$ and $^{222}Rn$. Radon-222, present as a gas or in solution, has a half-life of 3.8 days.

Stabilization of the uranium tailings would minimize human exposures through inhalation and ingestion, but no simple stabilization procedures currently exist. Several promising sealant/attenuation techniques have been developed and are being tested (Hartley et al. 1980, Nelson et al. 1980).

The useful life of sealants is subject to a number of physical, chemical and biological factors. An important aspect with respect to long-term stability of sealants is disruption of sealant integrity following penetration by plant roots, digging animals, or erosion of the stabilization cover. Short-term (1 to 4 years) control of plant growth can be attained by incorporation of herbicides into or over the sealant, but chemical and microbial degradation of the herbicide will rapidly destroy its effective phytotoxicity.

A plant root barrier system may be needed to prevent roots from damaging the integrity of asphalt emulsion or multilayer clay sealants over U-tailings for much longer periods of time. PNL has acquired extensive experience in developing stable polymeric carrier/delivery systems (PCD systems) which can be applied to problems of biological uptake and transport of radionuclides at burial sites for low level radioactive wastes (Burton et al. 1978). Management alternatives designed to reduce radionuclide transport have also been evaluated (Cline et al. 1980).

The PCD system design should perform the following functions:

1) It must serve as a vehicle (polymer) that controls the release rate of the biocide into the soil near the barrier for a long period of time.

2) It must maintain an effective concentration of biocide in a narrow band of soil and limit migration into the rest of the root zone, thus allowing normal root growth above the barrier.

3) The selected biocide must have the ability to stop root elongation at the barrier only; no translocation to the remaining roots and above-ground plant parts should take place. It must be compatible with revegetation programs.

Burrowing animals have reached buried wastes. Radioactive salts were exposed by deep-burrowing animals (O'Farrell et al. 1975). The exposed salts were directly ingested by blacktailed hares, which spread $^{90}Sr$ and $^{137}Cs$ in fecal pellets and urine. After an asphalt coating was put down to reseal the source of salt, fecal pellets of the blacktailed hares were collected from the crib area and sorted according to age. The test results showed that the asphalt pad had probably sealed the source of contamination (Uresk et al. 1975). This incident indicated the need to develop biobarriers that would prevent animal intrusion into buried wastes. These biobarriers would have the added requirement of preventing breaching of the seals placed over U-tailings to reduce radon gas emission to the environment.

## 1.2 DESCRIPTION AND PURPOSE

The purpose of this study was to develop and test physical and chemical barriers that would prevent animal and plant penetration of radon seals placed over U-tailings. The requirements for these barriers were that they would not harm the seals, aboveground vegetation, or other biota of the area. The containment systems must be compatible with the U-tailing site and be effective as biobarriers to animal and plant root intrusion for extended periods of time (up to 1000 yr).

## 1.3 PROGRAM AUTHORIZATION

In April 1980, the research proposal entitled "Application of Long-Term Chemical Biobarriers for U-Tailings" was approved by the Uranium Mill Tailings Remedial Action Project Office, Department of Energy, Albuquerque, NM. The applicable B & R code was AH-10-15.

The PNL Field Task Proposal dated February 11, 1980 was used as the basis of the research and development effort for FY-1980 with some changes resulting from the specific recommendations developed in FY-1981.

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# 2.0 CONTROLLED RELEASE SYSTEMS

## 2.1 INTRODUCTION

A single application of a herbicide such as trifluralin results in much higher concentrations than those necessary to achieve the desired effect (Figure 2.1). With time (only a few months to a few years depending on conditions), however, the concentration of the herbicide placed in the soil will be reduced by physical, chemical and biological action to less than the minimum effective level (MEL). Using controlled release devices, the active ingredient (herbicide) can be maintained at effective levels for extended periods of time. Polymers serve as effective "packages" for trifluralin not only because they act as a reservoir and release-regulating mechanism for the herbicide, but also because they protect the herbicide from degradation.

Trifluralin contained in a polymer matrix diffuses to the surrounding environment at a rate determined by its solubility and rate of diffusion in the polymer matrix. By selecting a polymeric carrier/delivery (PCD) system which can contain a large amount of trifluralin and yet release it very slowly to the environment, the herbicide can be delivered for periods of 100 years or more.

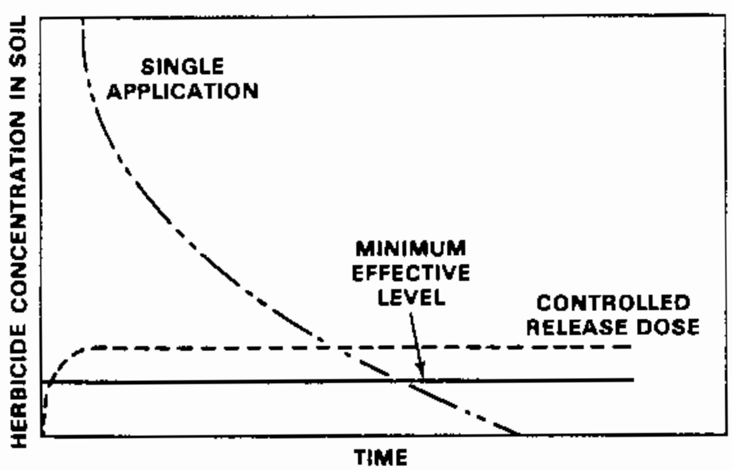


Figure 2.1. Comparison of Herbicide Levels Over Time for a Single Application and Controlled Release

The PCD system in this application has an element of feedback control, reducing the release of herbicide when a large concentration of herbicide is in the soil surrounding the device. Release of herbicide from the device is determined by Fick's Law, which defines the amount released as proportional to the difference in concentration of the herbicide in the device and at the surface of the device. By choosing a herbicide which is tightly bound to the soil in the area immediately surrounding the device, the amount released from the device will decrease as surface or soil concentration increases, since the concentration gradient across the diffusion distance will be less.

Controlled release of herbicides has not been studied intensively. The purpose of the few studies reported to date has been primarily to extend the lifetime of the herbicide in soil by only a few weeks. By using a microencapsulated formulation, Gentner and Danielson (1976) extended the herbicidal effectiveness of chlorpropham from 31 days (commercial formulation) to 66 days. Doane, Shasha, and Russell (1977) reported the results of a study by Schreiber (1976), which indicated the S-ethyl dipropylthiocarbamate encapsulated in starch xanthide extended the herbicidal action of this material by several weeks. Allan, Beer, and Cousin (1977) showed that 4.9% α-cellulose-2,4-dichlorophenoxyacetate (2,4-D) inhibits germination for 18 days, in contrast with free 2,4-D, which remains active for only nine days. McCormick and Fooladi (1977) have shown that controlled-release formulations of metribuzin attached to polyvinyl alcohol were phytotoxic for more than 122 days, while the metribuzin control was no longer phytotoxic at 78 days.

A significant exception to these short-term herbicide tests has been reported by Allan et al. (1980). They conducted a field trial using a controlled-release formulation of Douglas fir bark and 2,4-dichlorophenoxy-γ-butyric acid. In areas where deciduous brush was treated with this formulation, Douglas-fir seedlings grew significantly faster than untreated controls. At one application rate, the significantly accelerated yearly growth rate continued for at least five years after treatments.

## 2.2 DEVICE DEVELOPMENT

Controlled release systems or devices can be fabricated by a number of techniques, depending primarily on the demands of the application. One such system is the so-called "homogeneous" system, in which the active ingredient (the chemical to be released) is uniformly dispersed or dissolved in the polymer (reservoir) matrix. The device is prepared in a selected geometry, either by polymer extrusion or molding techniques in which the polymer and active ingredient are usually preblended. An additional technique allowing even more uniform release requires coating a device with a known thickness of additional polymer, which acts as a finite diffusion barrier.

In other instances, the release system consists of a tube or hollow cylinder whose interior acts as a reservoir for the active ingredient. The walls of the device, which act as the rate-controlling barrier, are of preformed materials. The reservoir is filled by a variety of techniques with the active material. Still another technique chemically bonds the bioactive ingredient to the polymer carrier structure, where it is slowly released by chemical or biological action.

These techniques use either synthetic or modified, natural polymers which, for the most part, can be fabricated by standard techniques and which act as reservoir, carrier, protective agent and release rate determinant for the active ingredient.

The studies reported here used materials in one of two shapes. The initial study materials, used to determine and compare release rates from a variety of polymers, were sheets made of homogeneous mixtures of the particular polymer and trifluralin. Table 2.1 lists the polymers that were studied as potential matrices in PCD devices. Later test materials were cylindrical pellets made from homogeneous mixtures of a variety of polymers and trifluralin. The devices prepared for use in the field trials combined a powdered polyethylene material, carbon black, and trifluralin, again in a cylindrical (pellet) shape.

Table 2.1. Polymer Types, Designation and Supplier

| Polymer Type | Designation | Supplier |
|---|---|---|
| 1. poly(ethylene-vinylacetate) (A) | Microthene 763 | USI Chemicals, New York, NY |
| 2. poly(ethylene-vinylacetate) (B) | Ultrathene UE-634 | USI Chemicals, New York, NY |
| 3. polyethylene (A) | Microthene MN 711-20 | USI Chemicals, New York, NY |
| 4. polyethylene (B) | Microthene MN 710-20 | USI Chemicals, New York, NY |
| 5. polyethylene (C) | Intramedic Medical formulation PHF 7480 | Clay Adams, Parsippany, NJ |
| 6. polypropylene (A) | Shell 7521 | Shell Chemical Co., Houston, TX |
| 7. polypropylene (B) | Profax 1600 | Hercules, Inc., Wilmington, DE |
| 8. poly(ether urethane) | Estane 5714-F | B. F. Goodrich Co., Akron, OH |
| 9. polyvinyl chloride | Geon 103EP | B. F. Goodrich Co., Akron, OH |
| 10. polyester | Hytrel 5525 | DuPont Corp., Wilmington, DE |
| 11. EPDM rubber (ethylene-propylene) | Nordel | DuPont Corp., Wilmington, DE |
| 12. silicone rubber (A) | Silastic 382 Elastomer | Dow Corning Corp., Midland, MI |
| 13. silicone rubber (B) | Silastic MDX4-4515 | Dow Corning Corp., Midland, MI |
| 14. silicone rubber (C) | Silastic Medical Grade silicone tubing | Dow Corning Corp., Midland, MI |
| 15. styrene-butadiene block copolymer | Kraton GX-6500 | Shell Chemical Co., Houston, TX |

## 2.3 SYSTEM FOR DETERMINATION OF RELEASE RATES

For a homogeneous device such as the one studied, the initial release rate from the PCD device is comparatively high until the trifluralin on and near the surface of the device has been released. High release rates permit rapid attainment of the trifluralin MEL in the region surrounding the device. Once surface trifluralin has been released to the soil, the release rate from the device then is determined by the distance the herbicide must diffuse to the surface of the device, the solubility of the herbicide in the polymer matrix, and the concentration of herbicide in the soil at the surface of the device. This last parameter determines the concentration gradient. For example, if the amount of herbicide in the immediate vicinity of the surface of the device is significant, herbicide will be released more slowly and the device will continue to release herbicide for a longer period of time than in the case where the concentration of herbicide at the surface of the device is maintained near zero. This effect will act to provide a constant concentration of herbicide in the area around the PCD device. In the absence of information on the concentration of herbicide at the surface of the device (and thus its effect on the release rate), it was decided to test the extreme case, in which the concentration of herbicide in the soil at the surface of the device is nearly zero. This will give an estimate of the minimum lifetime of the device. With additional information on the half-life of the herbicide in the soil to be used, it will be possible to determine the realistic lifetime of the device.

### 2.3.1 Testing Release Rates

To test release rates, the device (polymer in sheet form) was placed in a flow cell at the temperature of 13°C ± 1, a representative temperature in arid areas at about 1 to 2 m below ground level. Details of the flow cell used in determining herbicide release rates from sheets are illustrated in Figure 2.2; the flow cell itself, used in final tests of the pelletized PCD system, is presented in Figure 2.3. The extracting solution (water containing 10% methanol and 0.1% Triton X-100, to enhance the solubility of the herbicide) was pumped through the flow cell at a rate of 800 ml/day, and collected in a glass container. All lines exiting from the flow cell were glass or metal to avoid absorption that occurs with plastic lines. The herbicide was extracted from the flow cell effluent with 5 ml of chloroform. Approximately 4 g NaCl was added to the aqueous solution

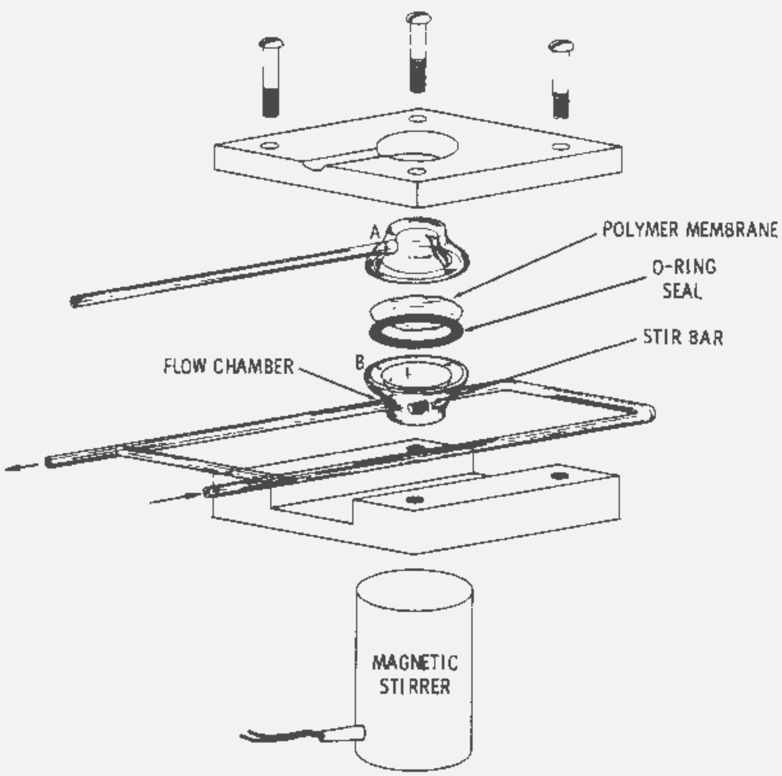


Figure 2.2. Details of Flow Cell for Determining Release Rates from Polymer Sheets

before shaking to achieve complete extraction of the herbicide by a salting-out effect. For each aliquot, the combined chloroform extract was brought up to volume in a 10 ml volumetric flask, and the concentration of trifluralin measured at 420 nm on a Cary 219 spectrophotometer. (The blank was a chloroform extract of fresh extracting solution.) The daily release rate was calculated from the concentration of herbicide in the aliquot, the length of time over which the sample was collected, and the volume of sample collected in that period.

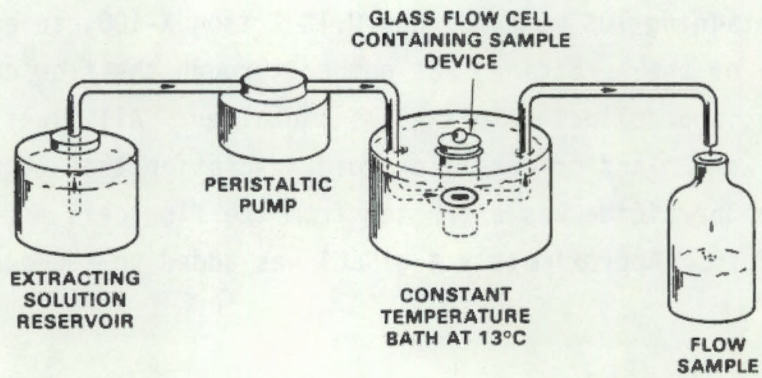


Figure 2.3. Continuous Flow System

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# 3.0 FEASIBILITY STUDY

## 3.1 INTRODUCTION

Laboratory feasibility studies revealed how controlled release herbicides at waste disposal sites would work. The tests were simple, making use of polymer membranes in sheet form and containing seven potentially active herbicides. Two common weed species, Russian thistle (Salsola kali L.) and cheatgrass (Bromus tectorum L.), were grown in polymethylmethacrylate lysimeters (approximately 30 in. deep) containing the herbicide-releasing membranes. Because no information was available on release rates of the selected herbicides from polymeric carriers, our choice of both polymer type and herbicide concentration was based on previous experience (Cline et al. 1982).

The first herbicide-containing membranes were prepared from a silicone rubber formation. Silicone rubber, a material used in earlier controlled-release studies, was expected to mix with and cure in formulations with the herbicide. The same concentration of each herbicide (10% by weight) was mixed with the silicone rubber because individual release rates were unknown for each herbicide. Seven herbicides were chosen for the initial study:

- trifluralin (α, α, α-trifluro-2,6-dinitro-N,N-dipropyl-p-toluidine)
- oryzalin (3,5-dinitro-N,N-dipropylsulfanilamide)
- DNBP (2,4-dinitro-6-sec-butylphenol)
- bromoxynil (3,5-dibromo-4-hydroxybenzonitrile)
- paraquat•Cl (1,1'-dimethyl-4,4-bipyridinium dichloride)
- bromoxynil ester (3,5-dibromo-4-hydroxybenzonitrile octanoic ester)
- TBA (2,3,6-trichlorobenzoic acid).

We anticipated that some of the herbicides, if released from the polymer membrane in sufficient quantities, would be translocated from the root, killing aerial portions of the plant. Other herbicides would prevent root elongation only.

## 3.2 EXPERIMENTAL PROCEDURES

To prepare herbicide-containing sheets, finely ground herbicide in solid form was thoroughly mixed with Dow Corning Silastic® 382 elastomer, producing a 10% concentration of herbicide with the uncatalized, viscous, liquid silicone rubber. The catalyst (stannous octoate) was added at approximately 1 drop per 10 g of silicone and thoroughly mixed with the silicone/herbicide blend. After degassing the blend by placing it in a vacuum system and pumping to greater than 29 inches vacuum for about 2 minutes, the fluid blend was poured between metal plates, pressed, and then cured at 60°C for about 20 minutes. Desired sheet thickness was obtained by placing appropriate shims around the plates.

The cured sheet containing a herbicide was removed from the plates and a disk 5 7/8 in. in diameter cut from each sheet. Three circular openings were cut in each disc (Figure 3.1), one (1 in. diameter) in the center, two others (1/2 in. diameter) on opposite sides of the center at points one-half the distance between the center of the membrane and the edge. The holes provided areas for root penetration and thus allowed us to test the effectiveness of the herbicide diffusion.

Membranes containing one each of the seven herbicides plus an additional control membrane with no herbicide were placed in soil in eight lysimeters approximately 18 in. below the soil surface. Russian thistle (*Salsola kali* L.) and cheatgrass (*Bromus tectorum* L.) were then planted in the lysimeters and placed in laboratory growth chambers.

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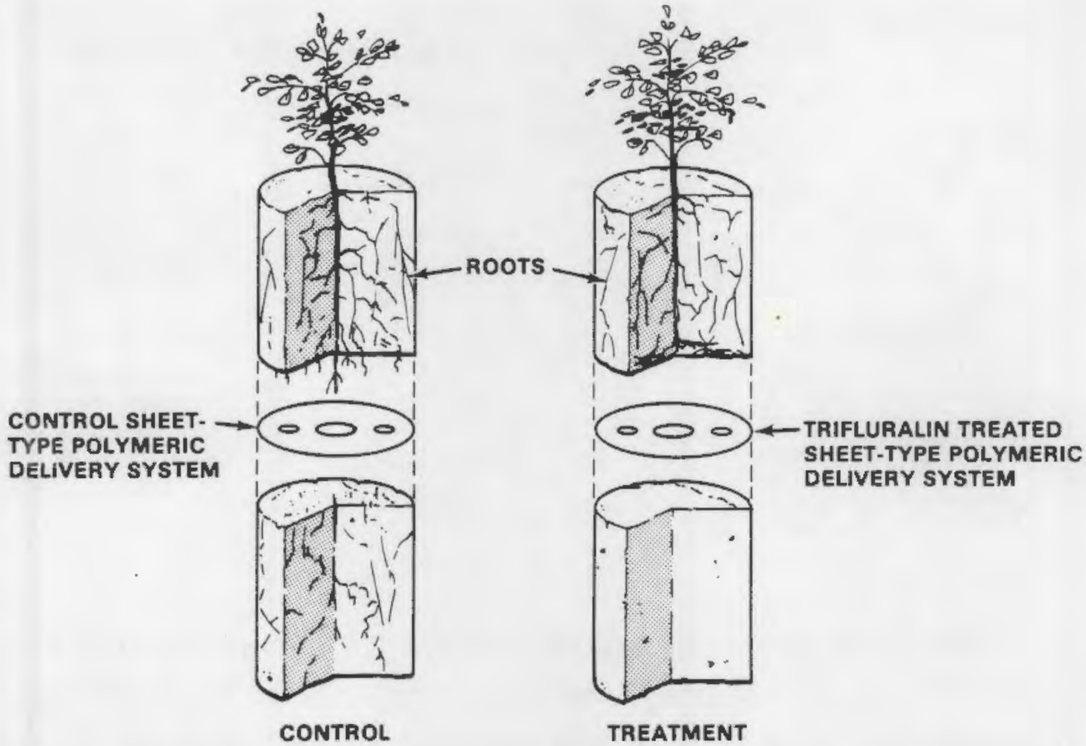


Figure 3.1. The Effect of Trifluralin-Treated Sheet on Root Elongation and Plant Growth

## 3.3 RESULTS AND DISCUSSION

After six weeks, plants in five of the eight lysimeters were dead or in poor condition, while plants growing in the control lysimeters and in the lysimeters containing trifluralin and oryzalin were flourishing. The lysimeters were sectioned and the plant root systems examined (Table 3.1). Roots penetrated the holes in the control membrane and also moved along the lysimeter wall past the membrane, reaching the bottom of the lysimeter. Plants growing in lysimeters containing TBA, bromoxynil and bromoxynil ester appeared dead or dying; oven dry weights for both above and below ground portions of these plants were lower than the control plants. In lysimeters containing paraquat and DNBP, roots grew past the herbicide barrier; shoot yields, in the lysimeters containing paraquat, however, were much lower than the control, and with DNBP, both root and shoot yields were very low (Table 3.1). Roots did not penetrate the herbicide barrier for either trifluralin or oryzalin, and in both cases shoot yields (oven dry weights) were similar to those of the control.

Table 3.1. Leaf, Stem and Root Weights (g/lysimeter) of Russian Thistle Grown in Lysimeters with Sheet PCD Systems.

| Treatment Number | Shoot Yield[a] (Leaf and Stem Tissue) Oven Dry Wt | Root Yield[b] Oven Dry Wt |
|---|---|---|
| 1. Trifluralin | 6.43 ± 0.96 | 4.75 |
| 2. Oryzalin | 5.44 ± 0.59 | 4.76 |
| 3. DNBP | 1.36 ± 0.10 | 1.54 |
| 4. Bromoxynil | 2.41 ± 1.75 | 0.53 |
| 5. Paraquat | 1.41 ± 0.14 | 5.48 |
| 6. Bromoxynil Ester | 1.68 ± 0.57 | 5.45 |
| 7. TBA | 1.44 ± 0.24 | 3.77 |
| 8. control | 6.61 ± 0.55 | 9.15 |

[a] $n = 3$, $\bar{x} \pm SE$, [b] $n = 1$

Of the seven herbicides tested, trifluralin appeared to show the most promise for application as a biobarrier. Roots in the lysimeter containing this herbicide approached no closer than 2 cm of the membrane. Moreover, the root mass in the soil above the membrane was very heavy and the plants appeared to be healthy.

## 3.4 TEST CONCLUSIONS

Our findings in this initial study support the conclusion that a polymer/trifluralin controlled release system would indeed stop root intrusion without destroying aboveground vegetation. Effects of the treated membrane barrier versus the control on root growth are depicted in Figure 3.1. Roots in a polymeric delivery system treated with trifluralin maintain their growth above the herbicide barrier and do not penetrate the barrier or soil beneath it. For these reasons, trifluralin was selected as the phytotoxin to be used in the field trial PCD system.

## 3.5 REFERENCES

Cline, J.F., D.A. Cataldo, W.E. Skiens and F.B. Burton. 1982. Biobarriers used in shallow burial ground stabilization. Nuclear Technology 58:150-153.

# 4.0 POLYMER SYSTEM STUDIES

## 4.1 INTRODUCTION

When work began on the uranium mill tailings biobarrier program in 1980, only the feasibility study using silicone rubber had been carried out (Section 3.0). Early data showed that trifluralin was released from silicone at a rate higher than that needed to prevent root elongation at the barrier. Other polymers appeared more promising as a carrier for the herbicide, since silicone rubbers are more difficult to process and more expensive than some thermoplastic polymers.

## 4.2 FABRICATION OF TRIFLURALIN-RELEASING SYSTEMS

As indicated in Section 3.0, polymers containing trifluralin were prepared in sheet form for the initial studies. Sheets prepared from most other polymers were made by melting to form a preblend of polymer and trifluralin between hot plates in a small laboratory press. Polymer sheets of poly(ether urethane) and poly(ethylene-vinylacetate) were made by casting a solution containing appropriate concentrations of the polymer and trifluralin to form a sheet. Tetrahydrofuran was used as a solvent for poly(ether urethane), and methylene chloride was used with poly(ethylene-vinylacetate). Sheets were generally prepared in the 0.010-0.020 in. range and cut to fit either in lysimeters or in the continuous flow test system (Section 2.3.1 and Figure 2.2).

Initial tests of a variety of polymer/trifluralin systems, along with evaluations of fabrication and application techniques, indicated that the most appropriate and economic form for production was as a flake or pellet. Our initial pellets were prepared by extruding a strand of polypropylene (B), containing trifluralin, through a small die. The die produced a strand about 2 mm in diameter, which was then chopped to produce pellets about 3 to 7 mm long. We found, after determining an experimental trifluralin release rate, that pellets of this dimension did not have sufficient reservoir capacity to provide the herbicide for more than one-third to one-half the desired period (100 years).

Further calculation and redesign based on data from greenhouse and laboratory studies indicated that a pellet about 9 mm in cross-section and

9 mm in length could provide the reservoir capacity necessary for a suitably long-lived device. In preparing pellets of this size for testing, a single-cavity aluminum mold was fabricated to be used in combination with a small, laboratory bench-scale injection molding machine. A small number of pellets of the desired size were prepared from a variety of polymer/trifluralin combinations. In addition to the blends of polymers and trifluralin in these pellets, other additives such as powdered silica and carbon black were added to the formulation. These additives were expected to have a number of beneficial effects on the PCD systems, including: increasing the reservoir capacity; improving the processing of the formulation into pellets; helping stabilize the polymer against degradation; aiding in the control of the trifluralin release; and decreasing the costs of the overall device.

After initial laboratory testing of these pellets, a formulation was selected to be used in preparing a preblend. Because neither extrusion dies nor chopping instruments were available to prepare pellets of this size (9x9 mm), a 24-cavity mold was fabricated. This mold, used on a 4 oz. fully automatic injection molding machine, produced the quantity of pellets needed for the field trials at the Grand Junction U-tailings Site.

Initial attempts to feed a mixture of polyethylene powder, carbon black (18 %) and trifluralin (24 %) were unsuccessful because the injection screw on the molding machine was unable to feed this mixture. A similar mixture of polyethylene pellets, carbon black pellets and ground trifluralin did not feed in the molder injection system either. Special preblends were then prepared by the following technique: Polyethylene powder (35 to 50 mesh) was mixed thoroughly with carbon black (18%) and the mixture warmed in an oven to 60-70°C. Trifluralin (melting point 48°C) was melted in a beaker and heated under agitation to 100-110°C. This molten trifluralin was then slowly blended into the warm polyethylene-carbon black mixture with constant mixing until cooled below the melting point of trifluralin. A friable black powder containing 24% trifluralin was obtained which readily fed in the injection molder screw system and permitted rapid molding of the PCD pellets. A mold cycle time of approximately 20 seconds was established and approximately 75,000 9 mm x 9 mm pellets were produced in a relatively short time. The majority of these pellets were then packaged and shipped to Grand Junction, Colorado for testing at the uranium mill tailings site. A number of these pellets were retained for testing and as controls in our laboratories.

## 4.3 RELEASE RATES OF TRIFLURALIN CONTROLLED-RELEASE SYSTEMS

Determination of the steady-state release rates of trifluralin from a variety of polymer sheets permits a preliminary evaluation of polymer suitability for use in the proposed application. The types of polymers tested and their commercial designation and supplier are shown in Table 2.1, while the release rates of trifluralin from formulations prepared from these polymers are shown in Table 4.1. In each case the polymer was loaded with approximately 10% trifluralin in a homogeneous mixture and a sheet formed to run in the continuous flow system until the release rate had reached steady state. Depending mainly on the polymer used, release rates varied over a 60-fold range, indicating the variety of rates that may be attained for this herbicide from only a relatively small number of the potential polymers available.

These initial studies used sheets because of the simplicity of determining release rates; however, for application in the field, pellets proved much more practical. Unlike pellets, sheets of polymer tend to tear

Table 4.1. Release Rates of Trifluralin from PCD Systems (Sheets, 10% Loading)

| Polymer Type | Release Rate<br>$\mu g/day/cm^2 \pm SD$ |
|---|---|
| poly(ethylene-vinylacetate) (A) | 9.3 ± 3.3 |
| poly(ethylene-vinylacetate) (B) | 13.1 ± 0.6 |
| polyester | 7.3 ± 1.7 |
| poly(ether urethane) | 3.4 ± 1.0 |
| polyethylene (A) | 1.5 ± 0.2 |
| polypropylene (A) | 4.2 ± 0.4 |
| polypropylene (B) | 3.9 ± 0.4 |
| silicone rubber (B) | 91.7 ± 16.8 |

when heavy equipment is used to cover the sheet with earth. Moisture may move freely through the soil profile when pellets are used, while films or sheets of polymer prevent free moisture movement. In addition, pellets are easier both to manufacture and apply.

A number of types of pellets were investigated for field application. Release rates for trifluralin from two types of tubing used are shown in Table 4.2. Trifluralin was mixed with silicone oil to maintain the mobility of the trifluralin in the reservoir. After plugging one end of the tubing, the mixture was then placed in the tubing and the open end sealed.

Tubing offers several advantages. First, the constant wall thickness of the tubing allows the release rate to remain constant over the lifetime of the device. Second, the method permits very high loadings of trifluralin in the device, and this extends the life of the device. The fabrication method for the devices would depend upon the particular polymer used. In the two cases shown in Table 4.2, the release rate for silicone rubber is unnecessarily high for most plant/weed species, while that for polyethylene is in the desired range. These polyethylene devices, containing approximately 230 mg of trifluralin, should release at the indicated level for 50 years. This particular approach was rejected for the initial field study due to the difficulty in fabricating large numbers of these devices with our available equipment.

In a second approach, pellets were fabricated, 1 to 3 mm in diameter and 2 to 7 mm long, from trifluralin-containing polymers by an extrusion process. Release rates for some of these devices are shown in Table 4.3.

Table 4.2. Release Rates of Trifluralin from PCD Systems (Tubing)

| | Release Rate | |
|---|---|---|
| Polymer Type/Size | μg/day ± SD | μg/day/cm$^2$ ± SD |
| silicone rubber (C) 4.65 mm OD x 3.35 mm ID 28 mm long | 124 ± 54 | 39 ± 17 |
| polyethylene (C) 4.83 mm OD x 3.75 mm ID 15 mm long | 12.7 ± 4.3 | 4.2 ± 1.4 |

Table 4.3. Release Rates of Trifluralin From PCD Systems (Small Cylindrical Pellets, 10% Trifluralin)

| Polymer Type | Release Rate, µg/day/g ± SD |
|---|---|
| Polyethylene (B) plus Novacite (silica) | 5.8 ± 1.9 |
| Polyethylene (B) | 15.9 ± 2.9 |
| Polypropylene (B) | 19.8 ± 2.4 |

Note that the addition of Novacite (a powdered silica filler material) in the polymer blend reduced the release rate of trifluralin from polyethylene pellets by a factor of nearly three. Since the surface of these pellets could not be measured accurately, release rates are reported per gram of pellet. A rough calculation indicates that the surface area of the pellets was on the order of 20 to 30 $cm^2/g$. This would indicate surprisingly low release rates as compared with other measurements made on sheets and larger pellets, a possible consequence of chance orientation of the polymeric molecules resulting from the extrusion process. At the release rates indicated, the trifluralin contained in the device would last for approximately 15 years. Actually, as the trifluralin is depleted from the device, it must diffuse further and further to reach the surface of the device. As a result, the actual release rate will decrease with time, and the life of the device will be extended.

At this point a decision was made to develop a large pellet and thus take advantage of an increased volume-to-surface area to provide increased herbicide capacity. After study of two larger geometries (Table 4.4), a pellet 9 mm in diameter and 9 mm long was selected for development of the initial field trials. This size, approaching a sphere, combines a relatively large available volume with almost the minimum possible surface area, particularly when compared to the small pellets and tubular devices discussed earlier. The size is also compatible with available equipment used to place seeds uniformly in soil. The loading of trifluralin was increased in these pellets to prolong the life of the device.

The results of some initial studies are shown in Table 4.4. Because sampling periods varied, release rates are not strictly comparable. In the last polyethylene sample, for example, release rates were determined on the same sample for two temporally distinct sampling periods. The samples used in this test were randomly selected from the large batch prepared for the field study. Previous samples (Table 4.4) were, in part, a study of the effect of carbon black on the amount of trifluralin which could be loaded into a polyethylene polymer. The carbon black appears to play no significant role on release rate as indicated by the first and second polyethylene samples in Table 4.4. Comparison of the second and fourth polyethylene samples indicates that increasing the loading of trifluralin (by 66%) increases the release rate also, but by a lesser amount (50%). As a result, heavier loaded devices should have longer lifetimes.

The release rate from controlled-release devices normally increases with increasing temperature. The nominal temperature for this study was 13°C, a typical soil temperature in arid areas 1 to 2 m below ground level. While that temperature is reasonably constant, the actual temperature encountered by the pellet is a function of the depth of placement. To determine the effect of temperature upon release rate, devices comparable to those used in the field trials were run at different temperatures in the continuous flow system. The devices were run first for 11 days at 37°C, then 14 days at room temperature (22°C), and then 24 days at 13°C. The release rates determined at these temperatures are given in Table 4.5. The release rate at 37°C is 30 times the release rate at 13°C; thus, a single day's release at 37°C is equivalent to a month at 13°C.

At the end of 49 days, the devices had released the amount of trifluralin that would have been released in 420 days at 13°C. Owing to interest in the release rate of trifluralin from these devices after several years of release, the temperature of the system was turned back up to 37°C and the system run for 54 days. During the final 18 days at 37°C, the release rate was determined (shown in Table 4.5 for five years). As expected, the trifluralin now had to diffuse for some distance through the device and the release rate was lower. Following this 54 day run at 37°C,

Table 4.4. Release Rates of Trifluralin from PCD Systems (Large Cylindrical Pellets)

| Polymer Type/Size/Loading | Release Rate (13°) µg/day ± SD | Release Rate (13°) µg/day/cm$^2$ ± SD |
|---|---|---|
| Polypropylene (B) (10 mm dia x 15 mm long) (7.4% trifluralin) | 21.6 ± 3.0<br>Sampling period; 12-48 days | 3.4 ± 0.5 |
| Polyethylene (B) (9 mm dia x 9 mm long) (14% trifluralin) | 10.6 ± 1.2<br>Sampling period; 185-195 days | 2.8 ± 0.3 |
| Polyethylene (B) (9 mm dia x 9 mm long) (13% trifluralin; 17% carbon black) | 11.1 ± 1.2<br>Sampling Period; 185-195 days | 2.9 ± 0.3 |
| Polyethylene (B) (9 mm dia x 9 mm long) (8% trifluralin; 26% carbon black) | 18.8 ± 4.1<br>Sampling period; 25-90 days | 4.9 ± 1.1 |
| Polyethylene (B) (9 mm dia x 9 mm long (18% trifluralin; 23% carbon black) | 16.6 ± 2.3<br>Sampling period; 160-170 days | 4.4 ± 0.6 |
| Polyethylene (A) (9 mm dia x 9 mm long) (22% trifluralin; 18% carbon black) | 1) 29.3 ± 2.0<br>Sampling period; 400-420 days<br>2) 13.1 ± 2.6<br>Sampling Period: 5 yr. | 7.7 ± 0.5<br><br>3.4 ± 0.7 |

the temperature of the bath was reduced to 13°C to allow determination the release rate for a device in operation (releasing trifluralin) for five years. This rate is also given in Table 4.5. The data suggest that release rates of devices subject to seasonal fluctuations in temperature benefit from them--that is, if the temperature around a device in the winter is lower than average, less trifluralin will be released (wasted). As the temperature increases during the growing season, increasing amounts of trifluralin are released, enhancing the possibility of preventing root elongation through the area of pellet placement.

Table 4.5. Release Rate as a Function of Temperature and Age of Device

| Temperature | Release Rate for New Device, μg/day ± SD | Release Rate for Device in Use Five Equivalent Years, μg/day ± SD |
|---|---|---|
| 37°C | 853 ± 91 | 198 ± 32 |
| 22°C | 131 ± 8 | -------- |
| 13°C | 29 ± 2 | 13 ± 3 |

## 4.4 LOADING OF TRIFLURALIN-RELEASING SYSTEMS

As we have already indicated, the lifetime of a PCD device increases with increased loading of trifluralin in the device, since release rate does not double with a doubling of the contained trifluralin. As a part of this research, sheets and pellets of various polymeric materials containing various concentrations of trifluralin were prepared. The solubility of the trifluralin in these polymer materials is limited, and thus the addition of silica and/or carbon black may increase the capacity of the polymer blend to hold trifluralin. Using polymers alone has led to immiscible mixtures of the polymer and trifluralin in many cases. As a result, even though the initial mixture contains a relatively high concentration of trifluralin, the final pellet often contains a much lower concentration and often a cooled and crystallized separate phase of trifluralin on the surface of the polymer (Table 4.6). The amount of trifluralin added in the original mixture is in the column labeled, "Theoretical," whereas the amount of trifluralin actually analyzed in the fabricated device is shown in the column labeled "Determined." Some polymers such as polyvinyl chloride incorporated the trifluralin easily; other polymers such as poly (ethylene vinylacetate) and poly(ether urethane) retain only relatively small amounts of the trifluralin. The addition of carbon black leads to retention of much higher amounts of trifluralin in some polyethylene devices and can be expected to increase the reserve capacity of many other polymer blends. Increasing the capacity also increases the longevity of the active device in the soil.

Table 4.6. Trifluralin Loading of PCD Systems

| Polymer Type | Carbon Black, % | Theoretical Trifluralin % | Determined Trifluralin % |
|---|---|---|---|
| Polyethylene (B) | None | 17 | 11 |
| Polyethylene (B) | None | 23 | 9 |
| Polyethylene (B) | None | 33 | 11 |
| Polyvinyl chloride | None | 17 | 9 |
| Polyvinyl chloride | None | 23 | 15 |
| Polyvinyl chloride | None | 33 | 33 |
| Styrene-butadiene Block Copolymer Rubber | None | 10 | 9 |
| Polypropylene (B) | None | 17 | 7 |
| Polypropylene (B) | None | 17 | 10 |
| Polyethylene (B) Novacite | None | 17 | 9 |
| Polyethylene (B) | None | 17 | 8 |
| Poly(Ethylene-vinylacetate) (B) | None | 23 | 10 |
| Poly(Ethylene-vinylacetate) (B) | None | 33 | 14 |
| Poly(ether urethane) | None | 23 | 6 |
| Polypropylene (B) | None | 17 | 12 |
| Polyethylene (B) | None | 23 | 14 |
| Polyethylene (B) | 18 | 19 | 16 |
| Polyethylene (B) | 24 | 18 | 12 |
| Polyethylene (B) | 21 | 26 | 24 |
| EPDM | 32 | 16 | 9 |
| Polyethylene (A) | 24 | 24 | 22 |
| Polyethylene (A) | None | 40 | 18 |
| Polyethylene (A) | 40 | 40 | 35 |

## 4.5 IN VITRO VERSUS IN SITU LIFETIME

The life of the device in vitro at 13°C may be calculated if the release rate of the device and the amount of trifluralin contained by the device are known. Since the device is homogeneous, requiring that the release rate decrease with time, there is no single release rate. We have, however, determined the release rate at the equivalent of five years, and this is 13.1 μg/day ± 2.6 SD. We can consider this to be a realistic average rate for a significant portion of the life of the device. The average device weighs 667.6 mg and contains 22.1% trifluralin, or a total of 146.5 mg trifluralin. At a release rate of 13.1 μg/day, the device would release trifluralin at a zero-order rate for 30.86 years. As indicated previously, this is the minimum effective life since 1) we are measuring the release rate in vitro at sink conditions where the concentration gradient is maximum, and 2) we are not taking into consideration the fact that the release rate will decrease somewhat with time.

To estimate what the possible life might be under actual conditions in the soil, we can incorporate the information available on the degradation rate of trifluralin in soil. The degradation rate of trifluralin in Ritzville silt-loam at 21°C and 18% moisture, at concentrations of 2 and 10 ppm, has been determined (See Section 5, Figure 5.1). The half-life of trifluralin under these conditions is ~50 days. (Note that the temperature involved is somewhat higher than the 13°C at which the trifluralinreleasing PCD system is placed. We will underestimate the effective life of the system as a result).

Using a half-life of ~50 days, we can calculate that approximately 1.38% of the trifluralin present is degrading each day. This is the amount that must be replaced each day by the controlled release device in order to maintain a constant level of trifluralin in the soil.

For 3 g of polypropylene (B) pellets, the in vitro release rate was shown to be 59.4 μg/day. When 3 g of these pellets was placed in 400 g of soil, the soil concentration increased from 3.25 to 4.38 μg/g soil over a 123 day period, for an average of 3.82 μg/g soil. This would be equivalent to 1.528 mg in the 400 g soil. Considering that 1.38% of this 1.528 mg (21.1 μg) would have to be replaced each day, the devices had to release this amount to keep the trifluralin concentration constant. Since the soil concentration actually increased over time, the devices released more than this minimum amount. The increased amount was 3.67 μg/day/400 g soil; therefore, the total amount actually released in situ was 24.77 μg/day. This would indicate that the in vitro release rate was 2.4 times higher than the in situ release rate, when trifluralin is present in the soil at 33.8 ppm.

If the device in situ releases at this lower rate, the amount of trifluralin remaining in the device in situ is greater, and the devices must be expected to release for a longer period of time; specifically, 2.4

times the length predicted by the in vitro data. Since the in vitro data predicted a 30.86 year life, incorporating the difference just cited would indicate that the actual life in situ is approximately 74 years.

Field data (Table 6.3) showed that the concentration of trifluralin in the soil was five times higher than the minimum effective concentration necessary to stop root elongation. Therefore, the release rate could be reduced by the addition of polymers to increase the life of the PCD system to at least 158 years.

As previously indicated, this is based on the release rate at approximately five years. In fact, the release rate will likely decrease with time, and the device will continue to release after 74 years, but at a lower release rate. We have purposely loaded and designed the device to release more than is necessary in the first years of its life so that the release rate in this later period is still sufficient to prevent root growth.

## 4.6 CONCLUSIONS

Based on our studies of the formation of and release rates from PCD systems, we conclude the following.

- The PCD system in pellet form is most desirable since it lends itself readily to machine application and doesn't interfere with water movement in the soil.
- A cylindrically shaped pellet 9 mm x 9 mm provided a sufficient trifluralin reservoir to remain active ∿100 years.
- Pellets installed in field studies were made from polyethylene powder mixed with carbon black (30%) and trifluralin (22%) pressed into pellets using a mold. Polyethylene was the selected polymer because its release rate of trifluralin was in the desired range.
- Release rates of trifluralin from the pellets increased with increasing temperature. The release rate at 37°C was 30 times the release rate at 13°C (soil temperature at 1 m below the surface).
- Half-life of trifluralin in the soil after release from the PCD system is 50 days.

## 5.0 PHYTOTOXICITY AND DEGRADATION STUDIES

Successful field performance of a PCD system requires that a minimal effective level (MEL) of phytotoxin be maintained in a narrow soil band for an extended period of time. This minimal level or concentration must be sufficient to restrict basipetal root growth and compensate for losses due to microbial decomposition, chemical degradation (hydrolysis) and dilution by diffusion out of the band of soil surrounding the PCD device. It is essential that phytotoxin concentrations in soils be minimized to eliminate ground water contamination or undue stress to plants employed in revegetation. In this research, we have sought to 1) determine soil concentrations of trifluralin required to inhibit basipetal root growth, 2) determine rates of microbial decomposition and chemical degradation; and 3) evaluate PCD devices for effectiveness under laboratory conditions (Burton et al. 1982).

### 5.1 EVALUATION OF EFFECTIVE TRIFLURALIN CONCENTRATIONS IN SOILS

After exploring a number of rapid screens, a small laboratory-scale lysimeter system was selected for testing our device. These lysimeters are 35 cm tall and 5 cm in diameter, with provisions for placement of a 2 cm band of treated soil or PCD device at 20 to 25 cm below the soil surface. Ports for sampling horizontal soil cores are provided at 2 cm intervals above and below the treatment zone.

Initial studies were undertaken to determine the MEL or concentration of trifluralin required in soil (Ritzville silt-loam) to inhibit root elongation of Russian thistle, a deep-rooted plant which readily invades disturbed areas. A 2 cm soil layer containing 0, 0.1, 0.4, 1, 10, and 20 ppm of trifluralin (μg trifluralin/g dry wt soil) was placed 20 cm below the soil surface, seeded, and plants maintained for 60 days. Soil was maintained at 18% moisture and plants maintained in controlled environment chambers with a day/night photoperiod of 16/8 hr and 22°/12°C.

Within 10 days following germination, roots penetrated the treatment zone at all concentrations below 2 ppm (Table 5.1). At concentrations of 2, 10 and 20 ppm, the maximum extent of root elongation (MRE) was 18, 15, and 15 cm, respectively. Analysis of soil cores at the depth of MRE showed

Table 5.1. Trifluralin Effects on Root Elongation and Shoot Dry Matter Production[a]

| Soil Concentration[b] (ppm) | Root Penetration[c] (cm) | Shoot Dry Wt. (g) | Trifluralin Concentration At Root Tip[d] (ppm) |
|---|---|---|---|
| 0 | Bottom | 0.99 ± 0.09 | 0 |
| 0.1 | Bottom | 0.80 ± 0.08 | NA |
| 0.5 | Bottom | 0.71 ± 0.04 | NA |
| 2.0 | 18 | 0.41 ± 0.06 | 0.36 ± 0.05 |
| 10.0 | 15 | 0.28 ± 0.02 | 0.47 ± 0.17 |
| 20.0 | 15 | 0.30 ± 0.07 | 0.52 ± 0.17 |

[a] Reps, $\bar{x}$ ± SD; Russian thistle used.
[b] Soils amended with trifluralin and 50 g soil placed in 2 cm band, 20 cm from soil surface.
[c] Depth of maximum root penetration from soil surface.
[d] Soil core removed at point of maximum root elongation, extracted and assayed for trifluralin concentration.

0.3 to 0.5 ppm of trifluralin to be effective in preventing root elongation. Although a significant reduction in shoot dry matter production was observed for all treatments, no toxicity symptoms such as chlorosis appeared; at least a portion of the dry wt reduction was a likely result of reduced rooting volume available for root growth imposed by the placement of the treatment zone. The results of these experiments set limits for release rates of early PCD systems under development.

Our studies were extended to include a series of 13 native plant species suitable for revegetation efforts in Colorado (Table 5.2). These were evaluated to determine MEL values and possible adverse effects on growth. Experiments were conducted using the laboratory scale lysimeters described earlier. Because of differences in plant growth rates and morphology, the duration of the studies varied from 31 to 59 days. Amended soil levels of 2, 10, and 20 ppm of trifluralin placed in a 2 cm zone at 25 cm below the soil surface were used. Root penetration was inhibited for all species studied. In general, significant reductions in shoot and root dry weight were not observed for the majority of plant species. Significant reductions were observed, however, for fourwing saltbush,

Table 5.2. Minimum effective Levels of Trifluralin Required to Inhibit Longitudinal Root Growth, and Effects on Shoot and Root Dry Weight[a]

| Plant | Time for Root to Reach Treated Zone (days) | Duration of Study (days) | Minimal Effect Concentration[b] (ppm) | Effect on Shoot/Root Dry Weight[c] (% of Control) |
|---|---|---|---|---|
| Russian thistle | 17 | 31 | 0.3 | 92/82 |
| Tansy mustard | 21 | 45 | 4.7 | 90/85 |
| Fourwing saltbush | 15 | 45 | 4.0 | 72/77 |
| Gardner saltbush | 16 | 45 | 3.1 | 115/94 |
| Winterfat | 18 | 55 | 1.9 | 57/50 |
| Crown vetch | 14 | 45 | 6.4 | 94/115 |
| Rocky Mtn. penstemon | 24 | 45 | 0.9 | 99/101 |
| Palmer penstemon | 20 | 45 | 1.5 | 102/97 |
| Whitmar wheatgrass | 13 | 45 | 0.8 | 97/85 |
| Thickspike wheatgrass | 21 | 59 | 0.7 | 71/67 |
| Russian wildrye | 14 | 56 | 0.5 | 86/82 |
| Lewis blue flax | 13 | 56 | 2.5 | 83/101 |
| Bitterbrush | 14 | 54 | 1.2 | 95/96 |

[a] Roots grew from 18 to 24 cm below surface; 2-cm treated zone located 25 cm from surface.
[b] Plugs for analysis removed from soil just below root zone.
[c] Mean of three replicates.

winterfat, and thickspike wheatgrass. It should be noted that with increased rooting volume, the previously observed reductions in shoot dry weight for Russian thistle are minimized. Based on the MEL observed for crown vetch (6.4 ppm), operating characteristics for PCD systems were upgraded.

## 5.2 MICROBIAL DECOMPOSITION AND CHEMICAL DEGRADATION

Effective use of a sustained release device such as the PCD system requires that the MEL be known for vegetation characteristic of a given site. Rates of losses resulting from chemical and biological degradation must also be known. This latter information is required for design of a PCD system; it is also important in establishing the effective diffusion zone from the PCD devices and represents an important aspect in limiting the amount of trifluralin that will leave the biobarrier zone and enter the environment (adjacent soils and/or waters).

When used as chemical biobarriers, PCD systems will be subjected to a range of subsurface conditions, including temperature and moisture extremes and oxygen tensions. The latter, whether predominantly aerobic or anaerobic, will influence the type of microbial decomposition which occurs. We determined the relative half-life of trifluralin in soils under aerobic conditions by amending a Ritzville silt-loam soil with 2 and 10 ppm trifluralin. These levels of trifluralin are within the range of the MEL found for various native species (Figure 5.1). The half-life of trifluralin at both 2 and 10 ppm was calculated at ~50 days for the 110 day incubation; half-life was determined for the parent compound using high pressure liquid chromatography. We could discern no influences of trifluralin concentration on half-life or on microbial turnover of the herbicide. As a result, studies were conducted using $^{14}C$ - labeled trifluralin amended to soils to separate effects of chemical degradation

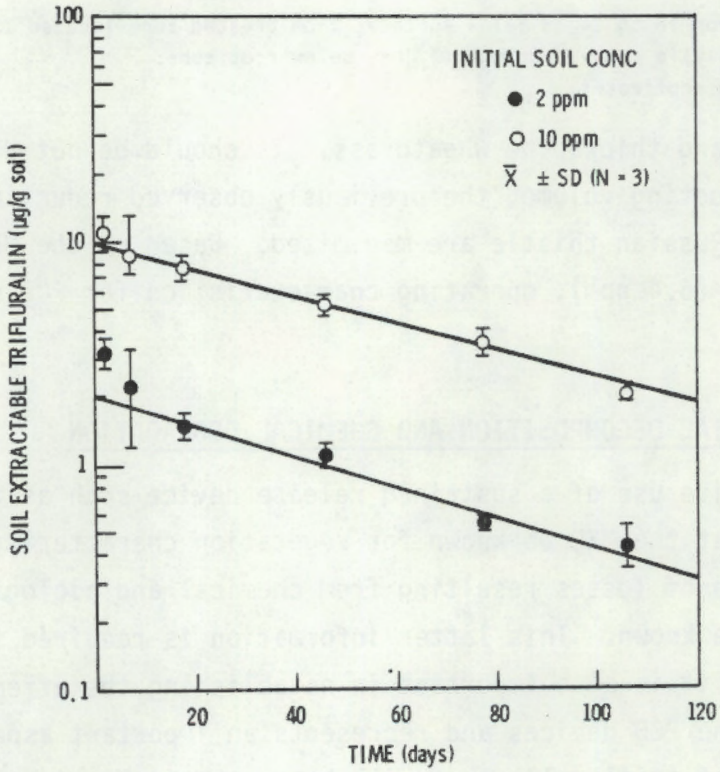


Figure 5.1. Degradation Rate of Trifluralin in Ritzville Silt-Loam, 18% Moisture, 21°C

processes from microbial decomposition processes. Experiments made use of "sterile" Ritzville and nonsterile soil with nutrients added to optimize microbial growth.

The latter mixture was incubated under either aerobic or anaerobic conditions to simulate deep placement conditions for PCD systems. The plots in Figure 5.2 show the concentration of extractable $^{14}C$-trifluralin over time for the three treatments. Here, soils were maintained sterile, or amended with nutrients and maintained under aerobic or anaerobic conditions. The half-life of trifluralin (10 ppm) in soils maintained under aerobic conditions was 31 days, compared to 119 days for soils maintained under anaerobic conditions. These should represent maximum decomposition rates. The decomposition rates (half-lives, Figures 5.1 and 5.2) calculated from these plots represent both microbial decomposition and chemical degradation of the trifluralin. An approximation was made of these two processes by following $^{14}C$-trifluralin losses from sterile soils lacking a significant microbial population (Figure 5.2). The half-life of

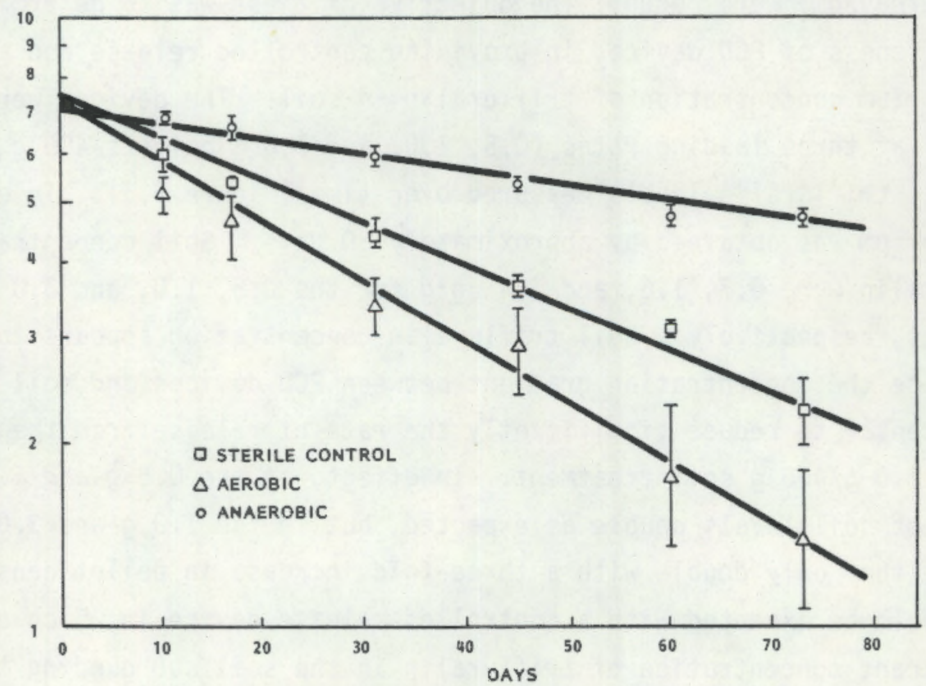


Figure 5.2. Effect of Microbial Growth on Trifluralin Persistence in Ritzville Soil

trifluralin, based on time of incubation, was calculated at 43 days. This indicates that approximately half of the trifluralin losses for aerobic soils (Figures 5.1 and 5.2) result from chemical degradation of trifluralin.

## 5.3 EVALUATION OF PCD DEVICES FOR EFFECTIVENESS UNDER LABORATORY CONDITIONS

The development of PCD devices progressed over a two-year period, and laboratory data for individual devices were employed to verify performance. Preliminary studies were conducted to test: 1) the suitability of a polypropylene (Profax® PS-1600) PCD device to maintain trifluralin concentration in soil; 2) the influence of soil moisture on the proposed Colorado PCD device; 3) equilibrium release rates for the Colorado PCD device in soil; and 4) the extent of diffusion gradients for single PCD devices.

Studies with the polypropylene (B) PCD devices (Table 4.6) with a functional lifetime of ~15 years (in vitro) and release rates of 19.8 μg/day/gm) were begun. The objective of these was to determine the effectiveness of PCD devices in providing controlled release and an equilibrium concentration of trifluralin in soil. The devices were placed in soil at three loading rates (0.5, 1.0, and 3.0 g pellets/400 g soil), and soil trifluralin levels measured over time (Figure 5.3). In each case, equilibrium was obtained by approximately 30 days. Soil concentrations of trifluralin were 0.7, 1.6, and 3.4 μg/g for the 0.5, 1.0, and 3.0 g loadings, respectively. Soil trifluralin concentration appears to influence the concentration gradient between PCD devices and soil and subsequently to reduce significantly the rate of release from the pellets in the 3.0 g/400 g soil treatment. In effect, at the 0.5 g and 1.0 g treatment soil levels double as expected, but in the 1.0 g and 3.0 g soil levels, they only double with a three-fold increase in pellet density. This would be expected with a controlled release device in which a significant concentration of trifluralin in the soil surrounding the device reduced the gradient and thus the release rate.

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In situ soil evaluation of the PCD device employed at the Grand Junction, Colorado field site was conducted using the laboratory scale lysimeter. Individual 9 mm x 9 mm devices (polyethylene A) were placed 25 cm below the soil surface at intervals of 2 cm and soil trifluralin levels measured over time (Figure 5.4). One lysimeter containing a single PCD device would simulate field placement at 2 in. intervals (36 pellets per square ft). Moisture was maintained at 12% during the study. The equilibrium soil concentration of 5.5 ppm trifluralin level was obtained after ~250 days, although 50% of this level was attained after 28 days of incubation. Perturbations in soil concentration observed between 28 and 100 days resulted from losses of trifluralin from the surface of the device and are expected, as noted in in vitro studies. Performance is also as predicted. It should be noted that these in situ studies were conducted at a quarter of the loading rate determined to be suitable for root control at

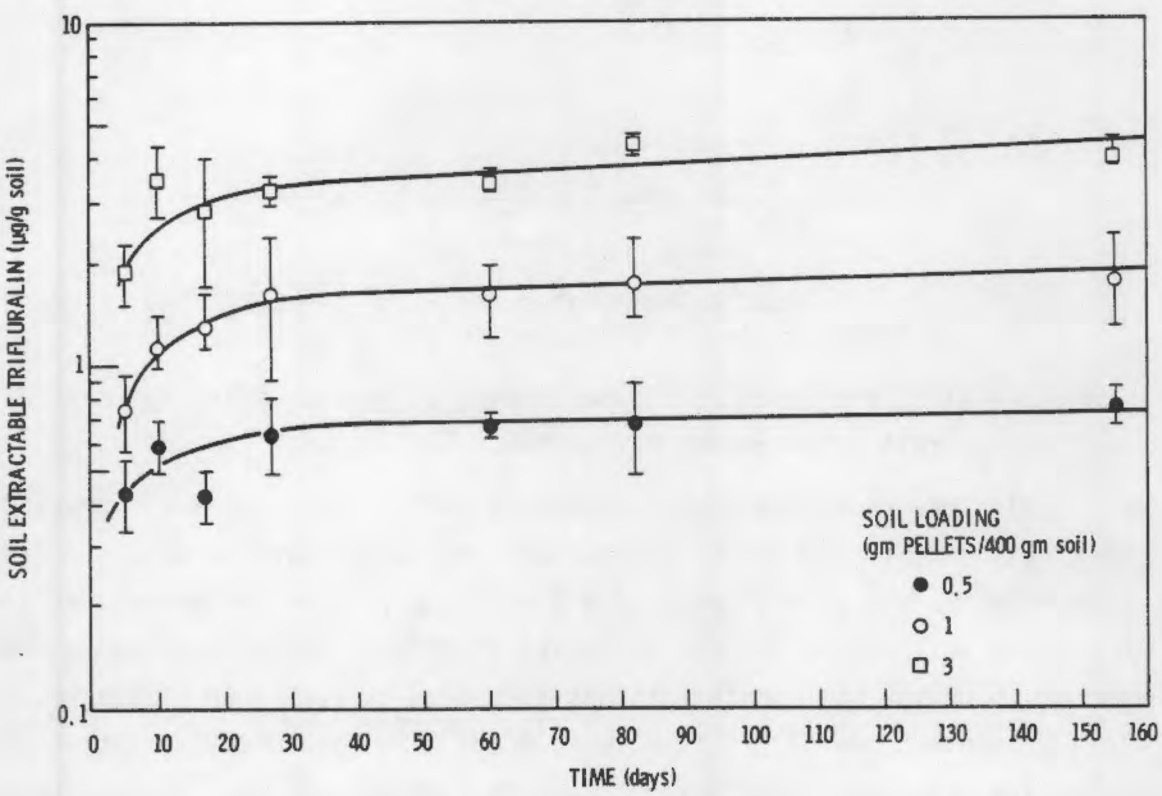


Figure 5.3. Trifluralin Concentration in Ritzville Silt-Loam with Time.

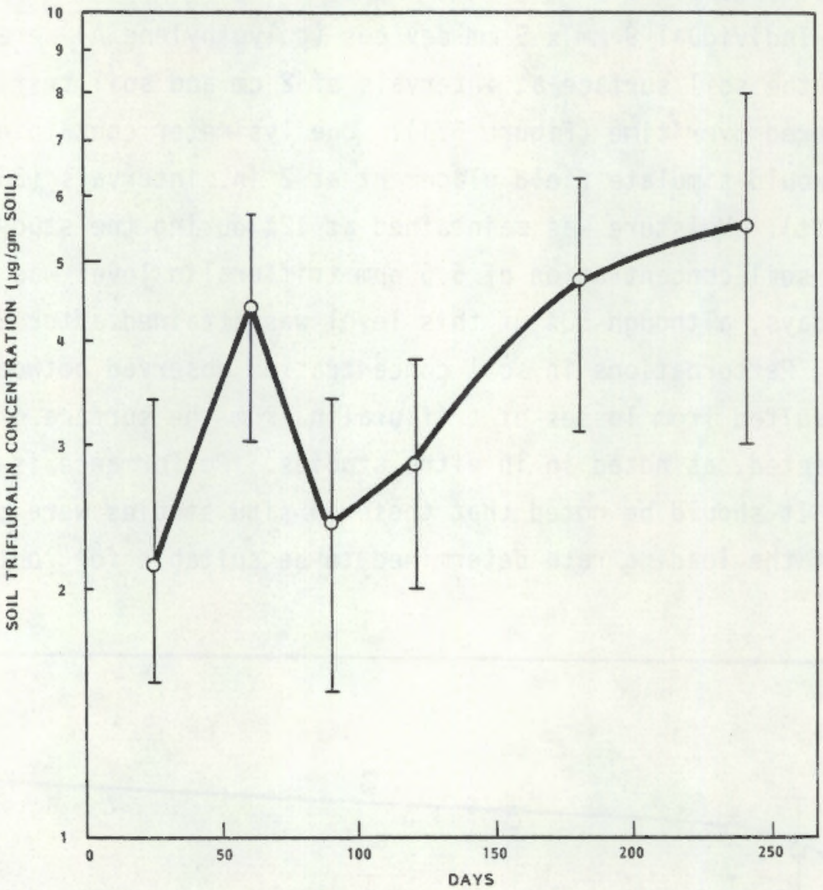


Figure 5.4. In Situ Soil Release Rates for 9 mm x 9 mm PCD Device (Polyethylene) in 900 g Soil Lysimeters.

Grand Junction, and based on this estimate, at no time after ~28 days did soil levels fall below needed MEL values.

Using the same laboratory lysimeters and PCD devices, the diffusion gradients established by the devices were measured over 250 days. In each case, the MEL was maintained over a 4 cm distance from the device; at 4 to 6 cm, the soil concentrations decreased from 20 to 60% of maximum values, and at 6 cm the soil concentrations decreased to ~10%. In most cases, soil concentrations of trifluralin at 6 cm, were substantially less than 1 ppm and less than detectable at 8-10 cm from the device. These data show the effective chemical barrier zone to be ~8 cm deep, with only minimal potential for environmental dispersal of trifluralin from the waste site.

An additional variable to be considered in estimating the effectiveness of PCD systems is their potential for seasonal changes in soil moisture levels. While soil moisture should have little detrimental effect on a PCD device, performance can be affected indirectly by alterations in the concentration gradient between the device and adjacent soil. Trifluralin is readily sorbed to soil particles, and this limits its diffusion away from the placement zone. Therefore, soil moisture and trifluralin solubility considerations can affect release rates from the device and also soil concentration levels. To evaluate this aspect of device performance, eight of the 9 mm x 9 mm devices were incubated in soils held at 6, 12 and 18% moisture. In effect, the presence of soil moisture reduces the capacity of soil to sorb trifluralin, saturates the diffusible pool surrounding the device, and reduces total device release. While soil moisture will affect the ultimate useful life of the PCD device, it isn't necessarily a detriment in the field, since plant roots will encounter higher trifluralin levels as the soil loses moisture.

## 5.4 CONCLUSIONS

Successful field performance of PCD systems requires that a minimum effective level of phytotoxin be maintained in a narrow band for an extended period of time. In these tests, a minimum effective level of trifluralin was maintained in a narrow band around the pellets so that longitudinal root growth beyond the pellet barrier was prevented. Minimum effective levels of trifluralin ranged from 0.3 to 6.4 ppm for 13 species of plants tested. Degradation of trifluralin by microbial and chemical processes was examined; the calculated half-life for this chemical was 50 days. Trifluralin concentrations in the soil around the pellets reached equilibrium ~30 days after placement. Examination of trifluralin diffusion gradients in the soil revealed that a minimum effective level of the herbicide was maintained 4 cm to 6 cm from the device.

## 5.5 REFERENCES

Burton, F. G., D. A. Cataldo, J. F. Cline and W. E. Skiens. 1982. The use of controlled release herbicides in waste burial sites. Proceedings of the Eighth International Controlled Release Symposium, Fort Lauderdale, Florida, July 26-29, 1981. (In Press).

## 6.0 FIELD AND GREENHOUSE TESTS

PCD systems that proved most promising in laboratory tests were selected for tests in the greenhouse or field plots. The objective was to determine if system performance under outside environmental conditions was as predicted from the laboratory tests. Release rates and equilibrium concentrations of trifluralin in the soil were measured around the PCD systems placed in lysimeters in a greenhouse, and in the overburden soils placed over U-tailings at Grand Junction. Visual inspection allowed us to determine if root growth stopped at the barrier.

### 6.1 TRIFLURALIN TREATMENT OVER ASPHALT EMULSION SEALS

This test was done to establish the effectiveness of trifluralin as a deterrent to root intrusions of asphalt emulsion seals. It was also necessary to test the placement of trifluralin relative to the seals to determine the best barrier placement against root elongation.

Two types of asphalt emulsion seals were placed in lysimeters made from PVC pipe 15 cm in diameter and 1 m long. One seal type contained a mixture of 20% asphalt emulsion and 80% sand, forming a plug in a hydraulic press. The resulting admix plug was 7.5 cm thick and 13.5 cm in diameter. The diameter of the plug was slightly less than the inside diameter of the lysimeter so that the plug could be slipped into position and asphalt cement poured between the sides of the plug and lysimeter to form a gas-tight seal. The seal was tested by placing helium under pressure below the seal (Figure 6.1). The second seal type was formed by pouring liquid asphalt emulsion over a soil layer approximately 0.9 m below the surface. The asphalt emulsion was allowed to harden before the upper layer of soil was placed in the lysimeters. After the asphalt seals were placed in the lysimeters, approximately 0.9 m of soil was added, watered to field capacity, planted to Russian thistle (<u>Salsola kali</u> L.) and placed in the greenhouse for a 90 day growth period. The different trifluralin/asphalt emulsion mixtures used in this test are shown in Figure 6.2. Each treatment was replicated three times for a total of 42 lysimeters.

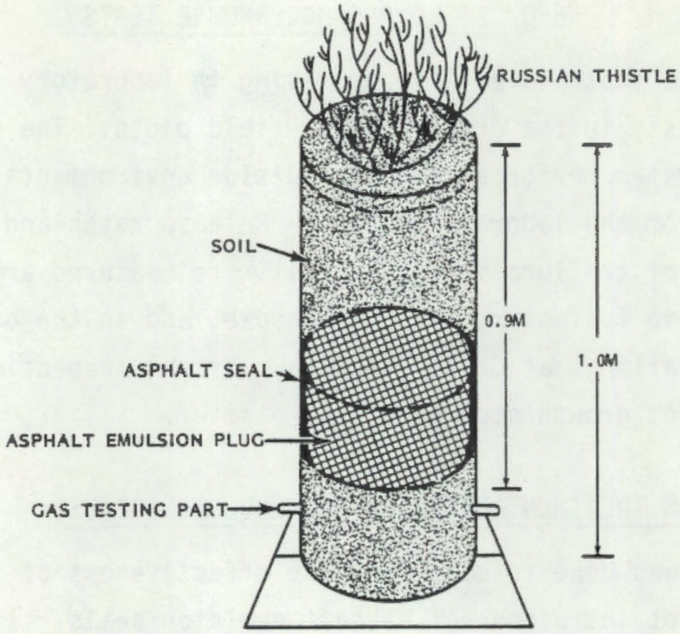


Figure 6.1. Experimental Design for Testing Trifluralin Treatment over Asphalt Emulsion Seals

After the 90 day growth period, the lysimeters were removed from the rack and split lengthwise by cutting a groove on opposite sides with a circular saw. When one-half of the lysimeter was removed, the relationships of root elongation to the seals were observed and photographed.

We observed the following:

- The asphalt emulsion plug, if properly installed, prevented root passage regardless of treatment.
- Plant roots in controls (no trifluralin) grew to the plug (Figure 6.2A).
- Trifluralin sprayed on to the plug prevented roots from contacting the plug surface directly (Figure 6.2B).
- Plant roots stopped over 1 in. above plug surface where the trifluralin was placed in the soil profile 1 in. above the plug (Figure 6.2C).
- The effectiveness of trifluralin as a root barrier was negated when it was mixed directly with the asphalt emulsion (Figure 6.2D).
- Plant roots elongated past the asphalt barrier (herbicide mixed into the emulsion) poured on in a thin layer (Figure 6.2E).

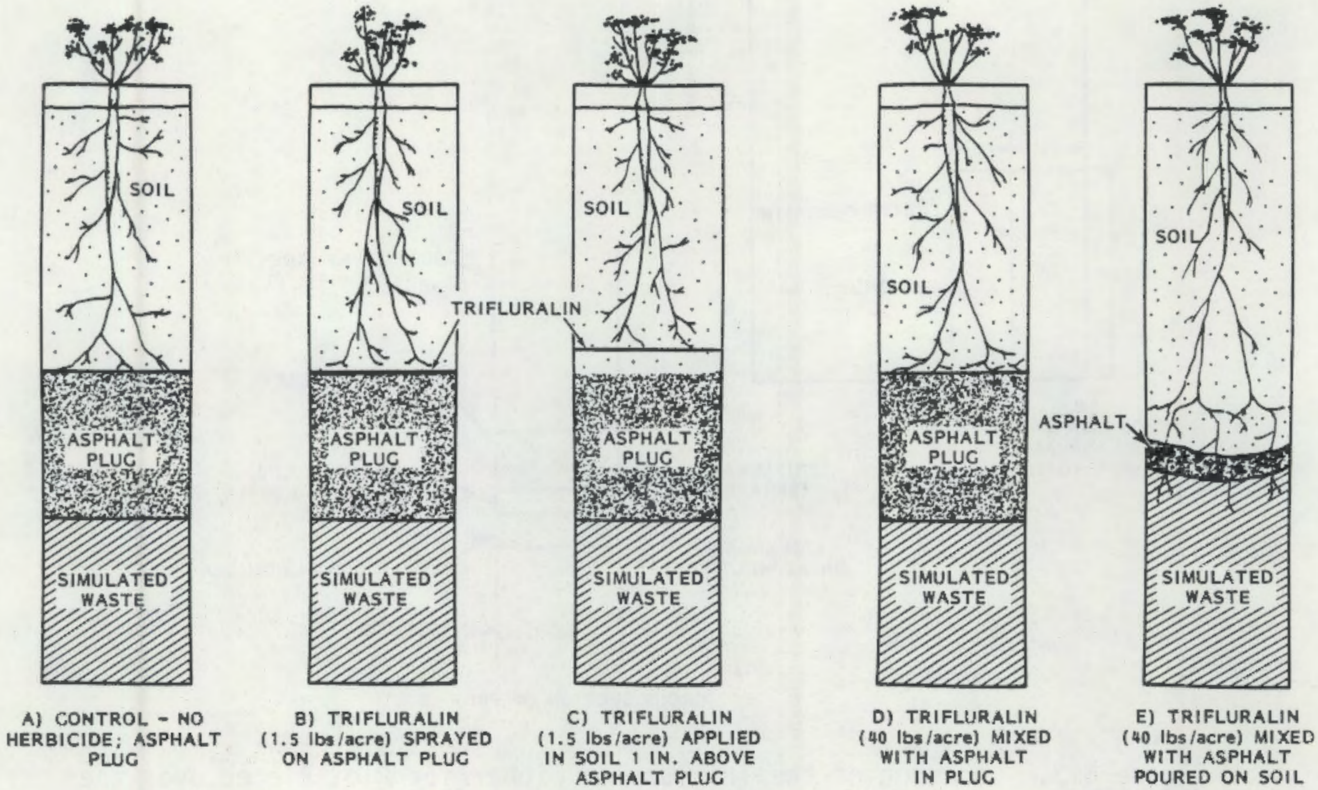


Figure 6.2. Effect of Trifluralin Placement (in relation to asphalt Seal) on Plant Root Growth.

## 6.2 LIQUID TRIFLURALIN TREATMENT OVER RADON SEALS AT GRAND JUNCTION, COLORADO (1980)

In August, 1980, trifluralin was applied with a hand spray wand to a section of the asphalt emulsion seal placed over uranium tailings (Figure 6.3). In a second treatment, lithium chloride was sprayed directly on to the asphalt emulsion seal. After spraying, we placed a 2 in. layer of sand over the lithium-treated layer. We then covered the sand with a 6 in. layer of soil and sprayed liquid trifluralin, at a pressure of 80 psi, on the top of this soil layer. Our application rate was 10 lbs of trifluralin per acre, a rate five to 10 times greater than those used in pre-emergence applications for one year's weed control in agricultural crops. Three separate spraying passes were made over the treated area to insure complete coverage.

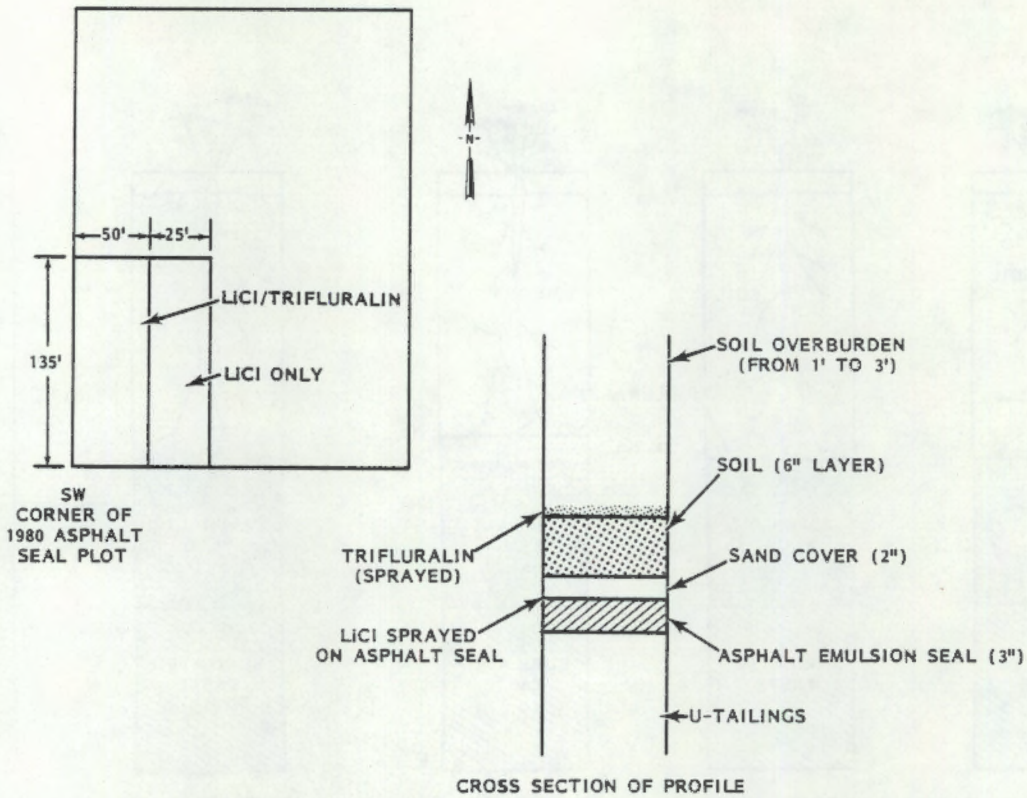


Figure 6.3. Location of the Trifluralin Biobarrier Plot Placed over the Asphalt Emulsion Seal, 1980.

Topsoil was placed immediately over the area treated with trifluralin to prevent degradation of the chemical from the sun's ultraviolet rays. Depth of this soil cover varied from ~3 ft on the north edge of the plot to 1 foot at the southern edge of the plot. Because lithium chloride was placed below the trifluralin layer in the soil strata, aboveground plant parts could be analyzed for lithium to determine if roots had penetrated the trifluralin layer and entered the lithium-treated layer.

Concurrent with our studies of trifluralin in conjunction with the asphalt emulsion seal, we conducted a similar test using a multilayer earth seal. Procedures for these experiments were the same, except the trifluralin and the LiCl tracer were sprayed on to the soil using the spray boom attached to the spraying machine instead of the hand wand. The same spray pressure of 80 psi and application rate of 10 lb trifluralin per acre were used. Again, three passes were made over the areas sprayed to insure complete soil coverage. The location of the test plot and vertical placements of the various components in the soil profile over the seal are shown in Figure 6.4.

Soil samples were taken in August of 1981 from a hole dug through the soil profile to the asphalt emulsion seal. These samples, removed along the vertical walls of the hole at various depths from the soil surface to the seal, were returned to the laboratory and analyzed for trifluralin content. A determination was made on the distance trifluralin had moved in the soil from the layer where it had been applied the previous year. In addition, we tested concentrations of trifluralin in the soil to see if they remained high enough to stop root elongation through the root barrier one year after application. We examined root patterns in the soil profile visually to determine if the trifluralin applied directly to soil layer two feet below the soil surface was actually stopping root elongation at the barrier.

The results (Table 6.1) show that roots stopped at the barrier 5 to 6 in. above the asphalt emulsion seal. Trifluralin concentration at this level was 6.45 ppm, sufficient to stop root elongation as found in laboratory studies (Table 5.2). Direct application in the soil profile would be effective as a root barrier for very short periods of time at best.

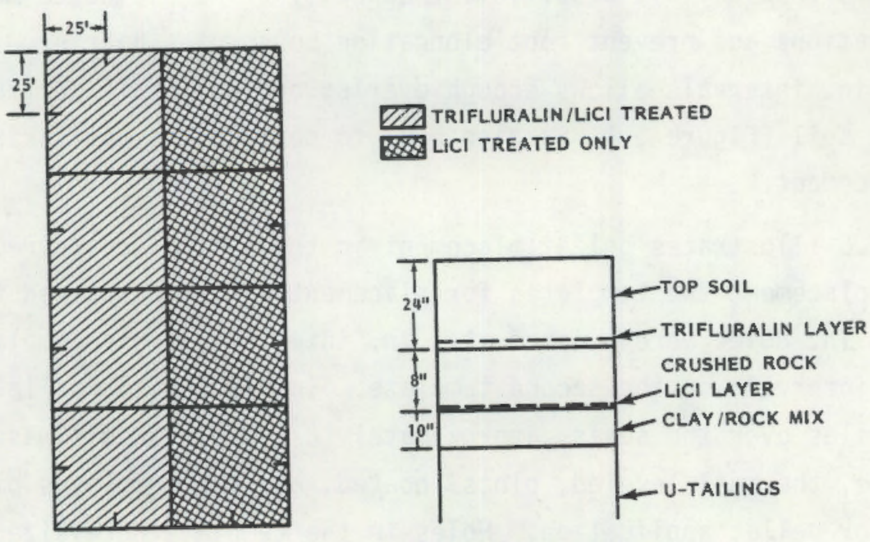


Figure 6.4. Location of the Trifluralin Biobarrier Plot Placed Over Multi-layer Earth Seal, Installed 1980.

Table 6.1. Trifluralin Content in Soil at the Barrier After One Year

| Distance Above Seal (in.) | ppm Trifluralin/g soil |
|---|---|
| 5 to 6 | 6.45 |
| 4 to 5 | 1.45 |
| 3 to 4 | 0.3 |
| 2 to 3 | 0.09 |
| 1½ to 2 | 0.15 |
| 0 to 1½ | 0.17 |

## 6.3 TRIFLURALIN TREATMENT USING PELLETIZED PCD SYSTEM AT THE GRAND JUNCTION TEST SITE

The PCD system consisting of manufactured pellets (described in Section 2) was placed over both the asphalt emulsion and multilayer earth seals constructed at the Grand Junction Test Site in August 1981. The pellets were placed on a 2 in. layer of soil above the seals. Two densities of pellets, 144 pellets/$ft^2$ (placed at 1 in. intervals) and 36 pellets/$ft^2$ (placed at 2 in. intervals), were applied to the multilayer earth seal. Pellets were placed over the asphalt emulsion seal at a density of 36 pellets/$ft^2$ only. Each of the three plots contained 200 $ft^2$; their locations are shown in Figure 6.5.

Greenhouse and laboratory studies indicated that pellets placed at 2 in. intervals should yield a sufficient quantity of herbicide to maintain soil concentrations and prevent root elongation beyond the barrier. Spacing at 2 in. intervals allows enough overlap of trifluralin diffusion (4 cm) in the soil (Figure 5.4, Section 5.3) to correct for any variation in pellet placement.

Figure 6.6 illustrates pellet placement in the soil. To insure precision of placement the templates for placement were constructed from Masonite; 5/8 in. holes were punched at 1 in. intervals on one template, and at 2 in. intervals on the second template. In placing the pellets in the soil profiles over the seals, approximately 2 in. of topsoil was placed over the seals, the soil leveled, plots located, and the templates placed on the soil for pellet application. Holes in the template were sized to hold only one pellet. The pellets were poured on to the template, then distributed individually by gently brushing the pellets into the holes with

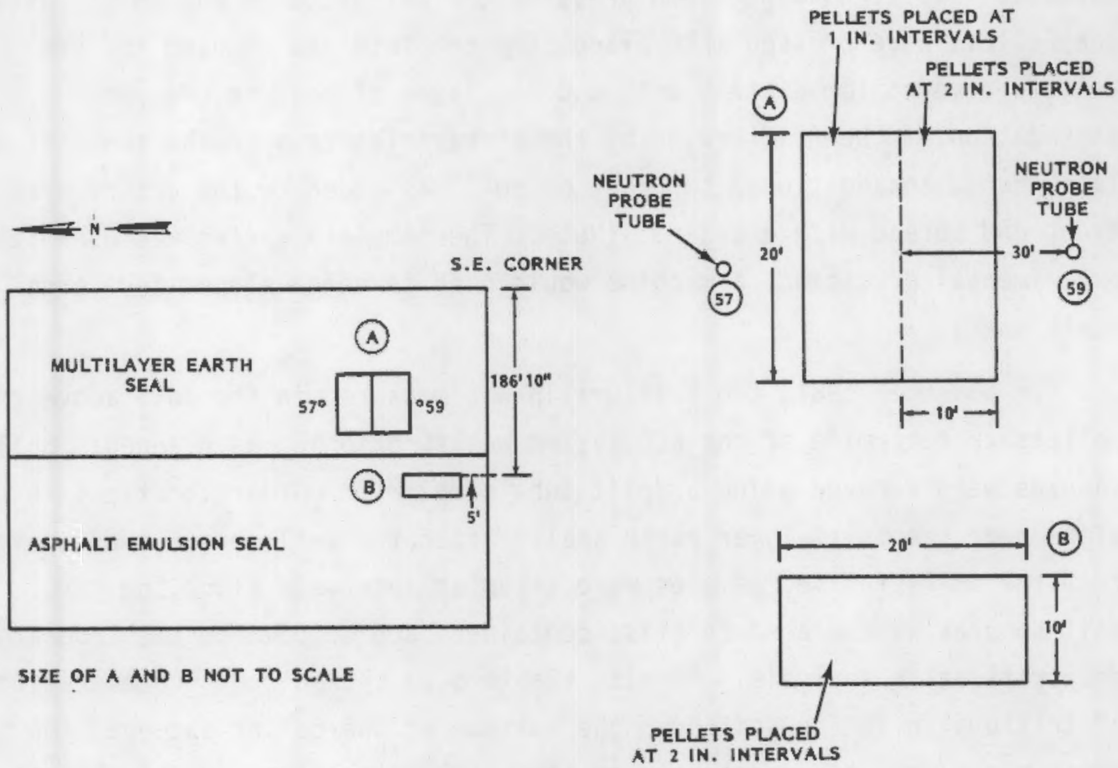


Figure 6.5. Location of Root Barrier Test Plots Placed over the Asphalt Emulsion and Multilayer Earth Seals, 1981.

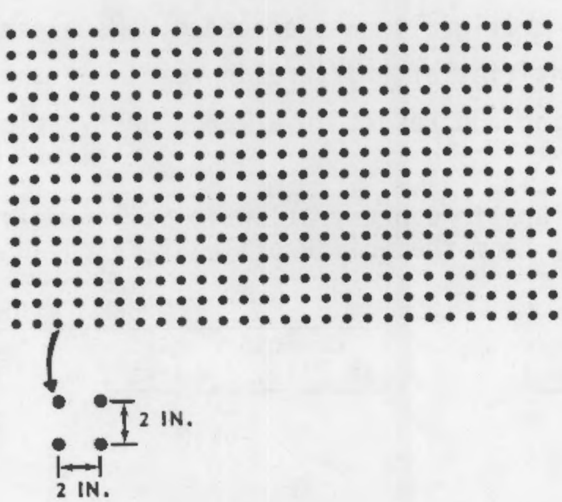


Figure 6.6. Placement of Pellets in the Soil Over Radon Seals at Grand Junction, CO, 1981.

a brush. Pellets were then pressed into place by placing a board over the template, applying weight, and pressing the pellets into the soil. After the pellets were pressed into place, the template was removed and the pellets covered immediately with a 6 in. layer of soil to prevent degradation of the trifluralin by the ultraviolet rays of the sun. At a later date, an additional two feet of soil was added to the entire area by truck and spread with a dozer. (Note: The template system was used for experimental precision; a machine would need to be developed for large scale use.)

In November 1981, the trifluralin was measured in the soil above the pellets to determine if the PCD system was functioning as planned. Soil samples were removed using a split tube sampler from four locations in the plots over the multi-layer earth seal. After the soil cores were removed from the soil profile, samples were taken at intervals along the core. The soil samples were placed in glass containers and shipped to the laboratory for trifluralin analysis. Results (Table 6.2) showed that concentrations of trifluralin in the soil near the surface of the pellet exceeded the minimum concentration necessary to stop root elongation. Where pellets were placed at 1 in. intervals, the concentration was exceedingly high (12,000 ppm) because a pellet was inadvertently left in a closed tube between the time it was removed from its field position and time of analysis. These early results show that the minimum effective concentration of trifluralin was exceeded. We expected this, since considerable quantities of trifluralin remain on the surface of the pellet after manufacturing and result in a large early release to the soil

Table 6.2. Trifluralin Concentration in Soil Samples Removed (November 1981) from the Soil Profile at Grand Junction.

| Distance above Pellets (in.) | ppm Trifluralin: Pellets Placed at 2 in. intervals | ppm Trifluralin: Pellets Placed at 1 in. intervals |
|---|---|---|
| 0-1 | 20 | 12,000.0 |
| 1-2 | 2.3 | 52.0 |
| 2-3 | 0 | 260.0 |
| 3-4 | 0 | 4.0 |
| 4-5 | 0 | 0.1 |
| 5-6 | 0 | 1.0 |
| 6-35 | 0 | 0.0 |

(Section 2.3). Lower soil concentrations of trifluralin were found in samples removed from the same area in April 1982 (Table 6.3), after the pellets had been in the soil for eight months. This time period was adequate for trifluralin levels to reach equilibrium.

The concentration of trifluralin (Table 6.3) in the soil near the pellet barrier still exceeded the concentration (6 ppm) necessary to stop root elongation at the barrier after approximately eight months in the field. If this concentration is maintained, this PCD system needs to be modified to slow down release of trifluralin from the pellets and increase the effective life of the PCD system. This can be accomplished by a change in the polymer matrix. The PCD system is functioning as predicted; a concentrated quantity of trifluralin is maintained near the barrier layer and does not move into the soil profile. Therefore, a normal root zone can be maintained in the overburden.

Table 6.3. Trifluralin in Soil Samples Taken (April 1982) from the Soil Profile at Grand Junction

| Distance Above Pellets (in.) | ppm Trifluralin: Pellets Placed at 2 in. intervals[a] | ppm Trifluralin: Pellets Placed 1 in. intervals |
|---|---|---|
| 0-0.5 | 41.3 ± 20.2 | 81.6 |
| 0.5-1 | 19.1 ± 14.6 | 15.4 |
| 1-2 | 4.5 ± 2.2 | 3.8 |
| 2-3 | 3.8 ± 3.8 | 0.6 |
| 3-4 | 0.2 ± 0.2 | 0 |
| 4-5 | 0.1 ± 0.1 | 0 |
| 5-6 | 0.1 ± 0.1 | 0 |
| 6-30 | 0 | 0 |

[a] 3 replicates

## 6.4 CONCLUSIONS

The following conclusions may be drawn from greenhouse studies and field tests of the PCD system.

- The effectiveness of trifluralin was negated when this herbicide was mixed directly with asphalt emulsion. Trifluralin sprayed on or above the seal prevented roots from reaching the surface of the seal.
- Liquid trifluralin in form of the parent compound remained effective for at least a year after placement in a layer 24 in. below the soil surface.
- The pellet PCD system placed in the overburden at Grand Junction, CO is performing as predicted 8 months after installation. Trifluralin remains in a thin layer above the PCD system.

# 7.0 ANIMAL INTRUSION STUDIES

## 7.1 LITERATURE SEARCH

The first objective of animal intrusion studies was to identify possible animal intruders that might pose a threat to the integrity of uranium mill tailings piles following stabilization. A literature search allowed us to compile the distributions of deep-burrowing rodents and ants in relation to 21 mill tailings sites (Table A.1, Appendix A). We have included information on habitats and burrowing depths of several important rodent and ant species in our study; the information was compiled and issued as a letter report in August 1980, and has been included in this report as Appendix A.

A more detailed literature search which considered only deep-burrowing animals was continued (Gano and States 1982). The information gathered included burrowing depths, soil types, and habitats preferred, along with recommendations on possible designs of burial sites which would discourage habitation by the various deep burrowers.

## 7.2 ANIMAL TEST BARRIERS

The second objective of this task was to test various barriers against intrusion by ants and deep-burrowing rodents. The two rodent species selected were representative of deep burrowing members of their genera common in western states. The Townsend ground squirrel (*Spermophilus townsendii*) is a burrower of medium size with widespread distribution. The white-tailed prairie dog (*Cynomys leucurus*) is much larger and a more powerful burrower. Prairie dogs are common in many areas near uranium tailings sites and their presence has been documented at the Grand Junction, Colorado tailings pile.

To determine burrowing capabilities of the deep-burrowing species under consideration, we constructed barrier test facilities at the Hanford site in Richland, Washington and at the Grand Junction, Colorado tailings pile.

7.2.1 Hanford Barrier Facilities

Eight enclosures, each 10 $ft^2$ were constructed to test the effectiveness of coarse crushed rock and asphalt emulsion against burrowing activities of Townsend ground squirrels and white-tailed prairie dogs (Figure 7.1). Using hardware cloth (soldered wire mesh), we covered the top, sides, and bottom of the pens except for a section of four square feet in the corner of each. In this area, a plywood box measuring 4 ft long by 4 ft wide and 4 ft deep was placed flush with the ground surface. This was the only area in which the animals were allowed to burrow (Figure 7.1).

We tested three different barriers. Each barrier was placed in two of the eight enclosures; the remaining enclosures served as controls. The barriers were designed in conjuction with the testing of radon sealants. One barrier was a variation of the multilayer earth radon sealant. It consisted of a 6 in. layer of sand covered by a 6 in. layer of bentonite/clay mixture; it was topped with a 6 in. layer of 1 to 1½ in.

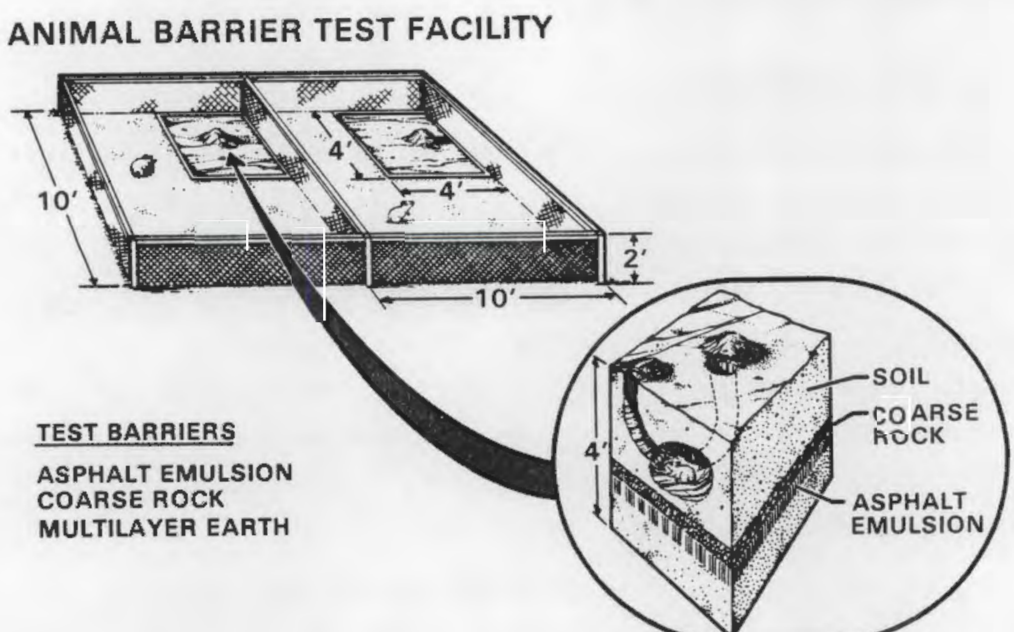


Figure 7.1 Design of Animal Barrier Test Facility

crushed rock (Figure 7.2a).The second barrier contained a 3 in. layer of asphalt covered with a 12 in. layer of 1 to 1½ in. crushed rock (railroad ballast), then 18 in. of topsoil (Figure 7.2b). The third barrier contained a 3 in. layer of asphalt emulsion covered with 18 in. of topsoil (Figure 7.2c).

A fine layer of pea gravel was placed over the crushed rock to prevent sifting of the overlying topsoil. Eighteen inches of soil was also placed over all barriers as a minimum of soil depth for burrowing and shelter. Both rodent species tested typically burrow much deeper than the 18 in. of topsoil provided here (Gano and States 1982); with such limited burrowing space, it was reasonable to expect the animals to challenge the barriers. The control boxes were used to demonstrate unobstructed burrowing within the confinement of the 4 ft box.

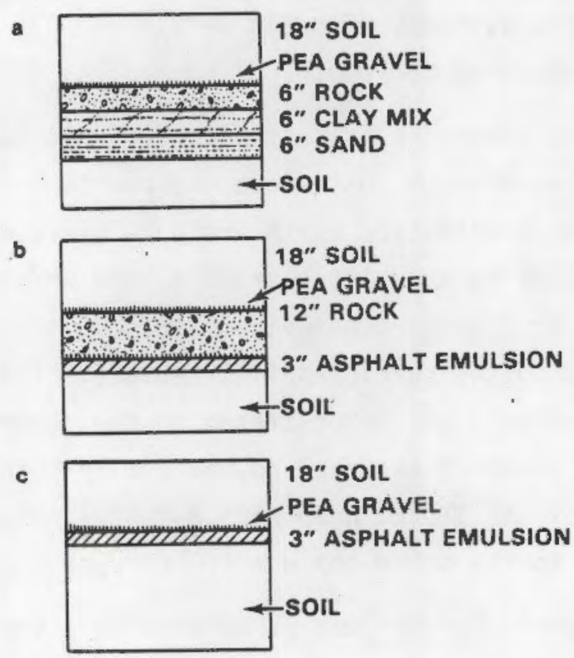


Figure 7.2. Barriers Tested

7.2.1.1 Ground Squirrels

During the summer of 1980, ground squirrels were added to each of the pens. They were quick to establish a tunnel for shelter in the burrow boxes. Food and water were provided as needed and burrowing activity noted. By September 1980, all squirrels except one had estivated and were no longer active; this animal remained active all winter. Aboveground activity resumed in January and February 1981. The animals were maintained in their pens eight weeks past the date when the last animal emerged from estivation. This provided sufficient time for construction and modification of burrow systems during the time of year these animals are most active. Upon removal from the pens, the animals were inspected for general health and released. All appeared healthy and had gained weight.

A backhoe was used to excavate a trench alongside the burrow boxes of each pen. By removing one panel of the boxes, we were able to expose the barriers and burrow systems. Tunnels were carefully excavated by hand, diagrammed, and measured for depth.

Tunneling was extensive in the soil above all barriers, with nest chambers in all cases at or just above the barrier. The two barrier types using crushed rock (multilayer earth and rock over asphalt) proved equally effective at preventing animal penetration, and while the burrow boxes over the asphalt barrier displayed no evidence of penetration, tunnels and nest chambers extended to the barrier-soil interface. The burrowing capability of the ground squirrels was demonstrated in the control boxes, where tunnels and nest chambers extended beyond 1 m in both cases. The nest chamber in one box was 110 cm deep with a tunnel extending to 122 cm, while the nest chamber in the other box was 100 cm deep.

The rock-asphalt barrier was penetrated by a single animal, ~5 cm only. The bottom of this nest chamber was ~4-5 cm below the level of the rock. Two other pens (one multilayer earth, one rock asphalt) containing rock barriers had no penetrations, although tunnels and nest chambers were excavated at the barrier-soil interface.

The deepest penetration into rock occurred in one of the multilayer earth barriers and extended approximately 12 cm. Another burrowing attempt in the same box extended approximately 4 cm into the rock. At excavation, the box had extensive tunneling; many tunnels were filled in with a mixture of soil and rock. This may represent an extreme case, since the pen contained a total of five squirrels. Lack of activity in February prompted the introduction of a female, who gave birth to four young.

#### 7.2.1.2 Prairie Dogs

Upon removal of the ground squirrels the pens and burrow boxes were reconstructed and white-tailed prairie dogs (Cynomys leucurus) introduced. Prairie dogs are roughly four times the size of the ground squirrels tested and are much more capable burrowers. Like the ground squirrels, the prairie dogs were maintained for a minimum of eight weeks. Accustomed to unlimited burrowing space, these animals thoroughly excavated the soil in the boxes.

The prairie dogs appeared able to move the rock much more easily than the ground squirrels. In the boxes containing multilayer earth barriers, the animals burrowed a maximum of ∿15 cm into the rock in both boxes, the approximate depth of the rock layer over the mixed clay layer. There were no open tunnels in the rock; however, loose soil with rocks and in one case, straw from nest material, indicated the animals had burrowed to that depth and then backfilled the tunnel. Apparently the animals did not disturb the wet clay layer in either box.

The animals in the pens containing the 12 in. rock layer over asphalt demonstrated a similar burrowing depth in the rock. In both of these boxes, tunneling extended 12-18 cm into the rock. The deepest penetrations in all rock layers occurred along the walls of the boxes, where digging may have been somewhat easier.

The asphalt barriers showed no evidence of penetration by prairie dogs. Because of extensive tunneling and backfilling, the soil was very loose in both boxes. In one of the two boxes we found few distinct tunnels; nearly half of the soil in the box was removed by burrowing efforts of the animal.

Burrowing by the animals was unhindered in the two control boxes. In one of these, the nest chamber was at 100 cm deep. Burrowing depth extended to 120 cm in the other box, and a tunnel extended at least 20 cm beyond the bottom of the box.

7.2.2 Grand Junction Barrier Facility

A barrier test facility was constructed on the Grand Junction tailings pile to test the burrowing capability of prairie dogs against a rock barrier using 1 to 1½ in. diameter round rock. Approximately 1 ft of washed and sized rock was placed over one corner of the experimental asphalt radon barrier, covering an area of 20 x 25 ft. The entire area was then covered with topsoil to a depth of 1 to 1½ ft over the rock. Three animal pens were constructed on this site with the walls of the pens extending below the soil and into the rock layer. Each pen provided an area 7 ft x 20 ft in which the animals were allowed to burrow. One animal was introduced into each pen.

A small hole was dug in the middle of each pen to encourage the animals to burrow there first. In all cases the animals utilized the holes and established burrow systems. The shallow soil forced them to the depth of the rock barrier, which caved in during burrowing attempts. The soil in each pen was extensively excavated until finally the animals were able to remove enough rocks near an edge or corner of their pen to tunnel out.

Round rocks of this size (1 to 1½ in. dia.) may not be satisfactory for preventing prairie dog intrusion in the long term. Sifting of soil and intrusion by plant roots could stabilize the round rocks and prevent cave in, allowing a tunnel to become established. Larger angular rock (crushed and sized) might provide a more formidable barrier. The 1-1½ in. crushed rock used in the barriers at the Hanford site appeared adequate for ground squirrels, but prairie dogs were able to penetrate to 15 cm. If a larger rock size were used that proved adequate for prairie dogs, it could be assumed adequate for all smaller animals.

7.3 CONCLUSIONS

In tests of animal intrusion at Grand Junction, CO and Richland, WA, ground squirrels did not penetrate any rock barrier, asphalt emulsion or multilayer earth seal. Prairie dogs, however, were able to penetrate a rock barrier constructed from 1 in. to 1½ in. crushed stone.

## 7.4 REFERENCES

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Appendix

# A LITERATURE REVIEW OF BURROWING DEPTHS AND HABITAT PREFERENCES OF BURROWING RODENTS AND ANTS

A literature survey was conducted to investigate rodent species that may present a threat to the integrity of radon sealants at uranium mill tailings sites. The focus of the survey was to identify species that may be present in the vicinity of these sites and to obtain information on burrowing strategies of the various species.

Geographic distribution of twenty prioritized tailings sites is shown in Figure A-1. The initial matching of these sites with species distribution maps (Hall and Kelson, 1959) resulted in the rough listing of "possible" species for each site shown in Table A-1. A perusal of this table reveals seven small mammal taxa which may have to be considered in approaches to stabilizing uranium mill tailings. In order of decreasing size, these taxa are 1) marmots, 2) prairie dogs, 3) ground squirrels, 4) pocket gophers, 5) kangaroo rats, 6) chipmunks and 7) pocket mice. Although microtine and cricetid rodents (such as *Microtus* in the east) could conceivably be important, we felt their likelihood of breaching biobarriers in a significant way was too low to warrant much attention now.

A large number of likely available species offers a wide variety of burrowing strategies, each of which may have special implications to mill tailings stabilization. The literature was searched to define the range of burrowing strategies represented in the present list, emphasizing the "extremes" which may be encountered. For example, we might learn that body size, soil texture preferences and average depth of frost penetration all interact to determine the depth to which animals tend to burrow. Table A-2 shows some recorded burrowing depths of mammals representative of the seven major rodent groups mentioned earlier. The badger (*Taxidea taxus*) was also included since it is an important consumer of small mammals, digging them from their burrows.

The black-tailed prairie dog (*Cynomys ludovicianus*) is the deepest burrower on the list (Table A-2). This animal will probably become one of the most important rodents to consider in developing a barrier to protect a radon seal over uranium mill tailings. It is a common inhabitant of several of the states in which mill tailings piles are located.

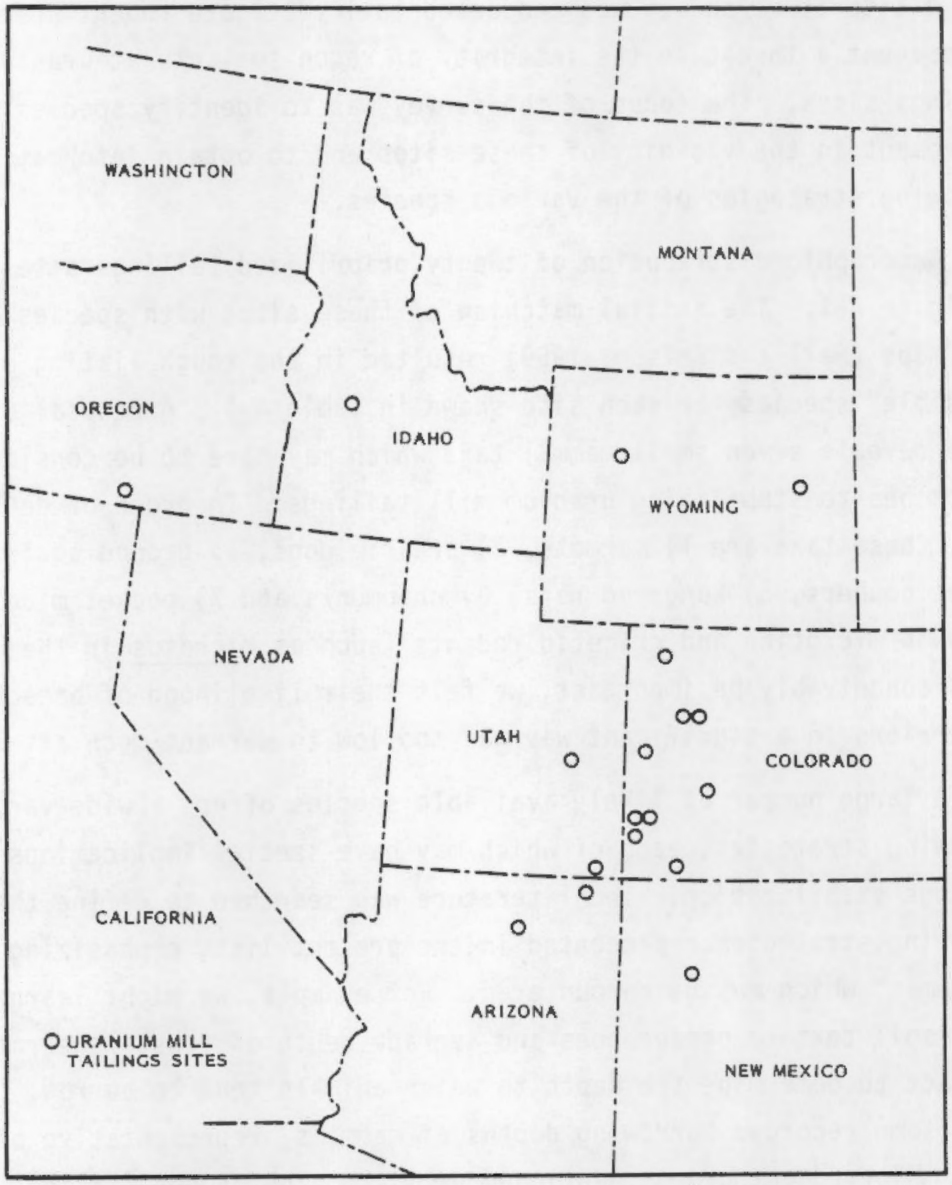


Figure A-1. Uranium Mill Tailings Sites

Table A-1. Rodent Species Whose Range Includes the Various Mill Tailings Sites

| Order Rodentia - Rodents | Mill Tailing Sites(2): High Priority | | | | | | | | Medium Priority | | | | | | Low Priority | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Salt Lake City, UT | Durango, CO | Shiprock, NM | Grand Junction, CO | Riverton, WY | Gunnison, CO | Old Rifle, CO | New Rifle, CO | Mexican Hat, UT | Lakeview, OR | Falls City, TX | Tuba City, AZ | Naturita, CO | Ambrosia Lake, NM | Green River, UT | Slick Rock (NC), CO | Slick Rock (UCC), CO | Maybell, CO | Monument Valley, AZ | Lowman, ID | Converse County, WY |
| **Family Sciuridae - Squirrels and Relatives** | | | | | | | | | | | | | | | | | | | | | |
| Eutamias minimus (Least chipmunk) | X | X | X | X | X | X | X | X | X | X | | X | X | X | X | X | X | X | X | | X |
| Eutamias amoenus (Yellow pine chipmunk) | | | | | | | | | | | | | | | | | | | | X | |
| Marmota flaviventris (Yellow-bellied marmot) | X | X | | X | X | X | X | X | | X | | | X | | X | X | X | X | | X | X |
| Ammospermophilus leucurus (White-tailed antelope squirrel) | X | X | X | X | | X | | | X | | | X | X | X | X | X | X | X | X | | |
| Spermophilus townsendii (Townsend's ground squirrel) | X | | | | | | | | | X | | | | | | | | | | X | |
| Spermophilus richardsonii (Richardson's ground squirrel) | | | | X | X | X | X | X | | | | | X | | X | X | X | X | | | X |
| Spermophilus armatus (Uinta ground squirrel) | X | | | | | | | | | | | | | | | | | | | | |
| Spermophilus beldingi (Belding's ground squirrel) | | | | | | | | | | X | | | | | | | | | | | |
| Spermophilus columbianus (Columbian ground squirrel) | | | | | | | | | | | | | | | | | | | | X | |
| Spermophilus tridecemlineatus (13-lined ground squirrel) | | | | X | X | X | X | X | | X | | | | | | | | | | | X |
| Spermophilus spilosoma (Spotted ground squirrel) | | | X | | | | | | X | | X | | | X | | | | | X | | |
| Spermophilus variegatus (Rock squirrel) | X | X | X | X | | X | X | X | X | | | X | X | X | X | X | X | X | X | | |
| Spermophilus lateralis (Golden-mantled ground squirrel) | X | X | | X | X | X | X | X | X | X | | | | | X | | | | | X | |
| Cynomys ludovicianus (Black-tailed prairie dog) | | | | | | | | | | | | | | | | | | | | | X |
| Cynomys leucurus (White-tailed prairie dog) | | | | X | X | X | X | X | | | | | | | X | | | | | | X |
| Cynomys gunnisoni (Gunnison's prairie dog) | | X | X | | | X | | | X | | | X | X | | | X | X | X | X | | |
| **Family Geomyidae - Pocket Gophers** | | | | | | | | | | | | | | | | | | | | | |
| Thomomys umbrinus (Southern pocket gopher) | X | X | | | | | | | | | | X | | | | X | X | X | X | | |
| Thomomys talpeides (Northern pocket gopher) | | | | X | | X | X | X | X | X | | | | | | | | | | X | |
| Geomys bursarius (Plains pocket gopher) | | | | | | | | | | | X | | | | | | | | | | |
| **Family Heteromyidae - Pocket Mice and Kangaroo Rats** | | | | | | | | | | | | | | | | | | | | | |
| Perognathus fasciatus (Olive-backed pocket mouse) | | | | | X | | | | | | | | | | | | | | | | X |
| Perognathus merriami (Merriam's pocket mouse) | | | | | | | | | | | X | | | | | | | | | | |
| Perognathus flavus (Silky pocket mouse) | | X | X | | | | | | X | | | X | | X | | | | | X | | |
| Perognathus apache (Apache pocket mouse) | | X | X | X | | | | | X | | | X | X | X | | X | X | X | | | |
| Perognathus parvus (Great Basin pocket mouse) | X | | | | | | | | | X | | | | | | | | | | X | |
| Perognathus hispidus (Hispid pocket mouse) | | | | | | | | | | | X | | | | | | | | | | |
| Perognathus intermedius (Rock pocket mouse) | | | | | | | | | | | | X | | | | | | | | | |
| Dipodomys ordii (Ord's kangaroo rat) | X | | X | X | X | X | | | X | X | X | X | | X | X | | | | X | | X |
| Dipodomys spectabilis (Banner-tailed kangaroo rat) | | | X | | | | | | | | | X | | X | | | | | X | | |
| Dipodomys merriami (Merriam's kangaroo rat) | | | X | | | | | | | | | | | | | | | | | | |

Table A-2. Burrowing Depths and Habitat Preferences of Some Representative Burrowing Mammals

| Species | Recorded Tunneling Depths (in cm) | Preferred Habitat | Soil |
|---|---|---|---|
| Marmota monax | 40-50 | Steep slopes | Well Drained Soils |
| Cynomys ludovicianus | 91-427 | Upland prairies | Fine textured |
| Spermophilus beecheyi | -- | Slopes w/ canopy | Fine w/ lrg. stones |
| Spermophilus townsendi | 50-80 | Grassland | -- |
| Spermophilus richardsoni | -- | Grassland | -- |
| Spermophilus tridecemlineatus | -- | Shortgrass prairies | -- |
| Tamias striatus | Louisiana 12.5-42<br>Wisconsin 61-86 | Upland hardwoods | Loessial |
| Eutamias minimus | -- | Sagebrush | -- |
| Eutamias amoenus | -- | Pinon pine | -- |
| Eutamias speciosus | -- | Lodgepole pine | -- |
| Thomomys bottae | 5-35 | Valleys to mt. meadows | loam to sandy |
| Thomomys talpoides | 10-30 | Prairies to pine forests | Fine |
| Geomys bursarius | $(\bar{x})$23 | Grasslands | Course, sandy |
| Pappogeomys castanops | $(\bar{x})$17; 132 max | Shortgrass prairies | Deep, sandy |
| Perognathus longimembris | 52-62 | Arid scrub | Fine or sandy |
| Perognathus parvus | 35-61<br>86-193 | Sagebrush<br>Chaparral | Fine or sandy |
| Dipodomys spectabilis | 40-50 | Arid scrub | Fine or sandy |
| Dipodomys microps | 24-45 | Arid scrub | Firm or sandy |
| Dipodomys merriami | 26-175+ | Arid scrub | Deep or Sandy |
| Taxidea taxus | 150+ | Grasslands | -- |

-- Information not yet received.

From the information gathered in this literature review, habitat preferences and natural histories of important burrowing animals have been summarized. This information will be used to develop barriers or create areas which are unattractive as habitats for burrowing rodents.

Ants are another important potential intruder. They are widely distributed and represented in nearly every conceivable habitat. Some species are capable of tunneling great depths. Table A-3 shows the tunneling depths of a few ant species, demonstrating the wide variety of tunneling strategies of these insects. Also shown is assumed habitat preference. Most of these species were collected in North Dakota and their habitat preference and tunneling depths may vary in different regions of the country (Wheeler and Wheeler 1963).

Table A-3. Tunneling Depths and Preferred Habitats of a Few Selected Ant Species

| Ant Species | Preferred Habitat | Maximum Recorded Depths of Chambers and Tunnels |
|---|---|---|
| Myrmica lobicornis fracticornis | No Preference | 8 cm |
| Manica mutica | Grassland | 69 cm |
| Pogonomyrmex occidentalis | Grassland | 310 cm |
| Pogonomyrmex owyheei | Grassland | 270 cm |
| Pheidole bicarinata vinelandica | Grassland | 8 cm |
| Pheidole pilifera coloradensis | Grassland | 10 cm |
| Monomorium minimum | Grassland | 6 cm |
| Solenopsis molesta | Grassland | 13 cm |
| Dolichoderus taschenbergi | Woods | 76 cm |
| Dorymyrmex pyramicus | Grassland | 122+ cm |
| Brachymyrmex depilis | No Preference | 70+ cm |
| Lasius sitkaensis | No Preference | 91 cm |
| Lasius flavus | Grassland | 46 cm |
| Acanthomyops interjectus | Grassland | 38 cm |
| Formica bradleyi | Sandy Soil | 15 cm |
| Formica (Proformica) limata | Grassland | 20 cm |
| Formica (Proformica) neogagates | Grassland | 61 cm |
| Formica (Raptiformica) obtusopilosa | Grassland | 51 cm |
| Formica (Raptiformica) rubicunda | Grassland | 15 cm |
| Formica (Raptiformica) sanguinea subnuda | No Preference | 15 cm |
| Formica obscuripes | Grassland | 30 cm |
| Formica ulkeri | Ecotone (Grassland/woods) | 30-152 cm |
| Formica fusca | No Preference | 109 cm |

From this list the deepest tunneling ants are the harvester ants, genus Pogonomyrmex. One of the most widely studied genera of ants, Pogonomyrmex was included in an annotated bibliography by Lavigne and Rogers (1974); 159 references are listed for the species occidentalis and owyeei. Sixteen species of harvester ants occur in the United States, most in arid regions west of the 95th meridian (Wheeler and Wheeler 1963). P. owyheei apparently has an affinity for disturbed soils (Rogers et al. 1978). If this is true for other species of Pogonomyrmex, this may be one of the most important genera to consider in waste management operations and remedial action on mill tailings piles.

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