Decomposition rates of residual crude oil in soil : a comparison of soil amendments
by Michael Francis Cormier

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Land Rehabilitation
Montana State University

Abstract:
Terrestrial spills of refined and crude oil adversely effect the productive capability of contaminated lands by inhibiting or eliminating vegetative growth. One practical approach to the correction of the problem posed by oil spills is to improve the conditions under which natural biodegradation of the spilled material takes place.

Ten different treatments were applied to soil contaminated with weathered crude oil at a site in north-central Montana using a combination of amendments including tillage, fertilization, irrigation, mulch, plastic sheeting, inoculum, and calcium chloride. Treatments were evaluated over a 10 1/2 month period. Pretreatment soil oil content in the 0 to 5 cm depth interval was 5.20 percent by weight, decreasing with depth to 1.40 and 0.41 percent oil by weight in the 5 to 15 and 15 to 46 cm intervals, respectively.

Tillage and chiseling were basic amendments applied to all treatments except the control. A significant reduction in soil oil content was noted (65%) in the 0 to 5 cm interval one day after their application, a result of the mixing of soil to a depth of 15 to 20 cm.

Following tillage, none of the treatments had significantly (P=0.05) higher degradation rates than the control over the term of the study. The combination of tillage, fertilizer, and calcium chloride exhibited the greatest decrease in soil oil content (22%), however.

This treatment also had significantly higher infiltration than non-contaminated soil as well as all other treatments. The increase in infiltration was attributed to flocculation of the dispersed sodic soils. Tilled plots also had significantly higher infiltration than non-tilled plots, suggesting that aeration and water flux is an important factor in oil degradation. Plots covered with plastic sheeting significantly increased soil temperatures above temperatures measured on non-covered plots at all depths but this increase did not influence the rate of degradation on plots covered with sheeting.

None of the other amendments were found to improve oil degradation. Two factors were primarily responsible for this result. The distribution of oil on the site was extremely variable, limiting the precision of the experimental design. In addition, soils were monitored for a relatively short period of time. In the cold, dry environment of the Northern Great Plains, even enhanced degradation of oil probably occurs at a slow rate.

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APPROVAL

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Michael Francis Cormier

This thesis has been read by each member of the thesis committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

January 6, 1989 — Date

D. J. Dollhopf — Chairperson, Graduate Committee

Approval for the Major Department

1-6-89 — Date

[signature] — Head, Major Department

Approval for the College of Graduate Studies

January 17, 1989 — Date

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ABSTRACT

Terrestrial spills of refined and crude oil adversely effect the productive capability of contaminated lands by inhibiting or eliminating vegetative growth. One practical approach to the correction of the problem posed by oil spills is to improve the conditions under which natural biodegradation of the spilled material takes place.

Ten different treatments were applied to soil contaminated with weathered crude oil at a site in north-central Montana using a combination of amendments including tillage, fertilization, irrigation, mulch, plastic sheeting, inoculum, and calcium chloride. Treatments were evaluated over a 10½ month period. Pretreatment soil oil content in the 0 to 5 cm depth interval was 5.20 percent by weight, decreasing with depth to 1.40 and 0.41 percent oil by weight in the 5 to 15 and 15 to 46 cm intervals, respectively.

Tillage and chiseling were basic amendments applied to all treatments except the control. A significant reduction in soil oil content was noted (65%) in the 0 to 5 cm interval one day after their application, a result of the mixing of soil to a depth of 15 to 20 cm.

Following tillage, none of the treatments had significantly (P=0.05) higher degradation rates than the control over the term of the study. The combination of tillage, fertilizer, and calcium chloride exhibited the greatest decrease in soil oil content (22%), however. This treatment also had significantly higher infiltration than non-contaminated soil as well as all other treatments. The increase in infiltration was attributed to flocculation of the dispersed sodic soils. Tilled plots also had significantly higher infiltration than non-tilled plots, suggesting that aeration and water flux is an important factor in oil degradation. Plots covered with plastic sheeting significantly increased soil temperatures above temperatures measured on non-covered plots at all depths but this increase did not influence the rate of degradation on plots covered with sheeting.

None of the other amendments were found to improve oil degradation. Two factors were primarily responsible for this result. The distribution of oil on the site was extremely variable, limiting the precision of the experimental design. In addition, soils were monitored for a relatively short period of time. In the cold, dry environment of the Northern Great Plains, even enhanced degradation of oil probably occurs at a slow rate.

INTRODUCTION

Terrestrial spills of refined and crude oil adversely effect the productive capability of contaminated lands by inhibiting or eliminating vegetative growth. Rowell (1977) estimated that an average of 69,000 barrels of oil was spilled each year in Alberta, Canada. In Montana, 150 incidents of petroleum spills were reported in 1987 (John Arrigo, personal communication, Montana State Department of Health and Environmental Sciences, Water Quality Bureau).

The effects of a spill may last for decades if left to natural processes (McGill 1977). In Montana, there are many oil and gas fields which harbor contaminated soils. Oil leaks and spills were common occurrences in the early years of oil exploration, a result of everyday production operations. Cleanup of these spills is difficult because a large proportion of them are small in size and are located in remote areas. In addition, responsible parties cannot often be identified due to changes in leasehold or land ownership. Removal of spilled oil, then, is left in the hands of the current landowner who has few resources to commit to the cleanup of contaminated land.

Although many researchers have investigated the degradation of crude oil in soils, no field studies have been conducted on highly weathered residual oils. The purpose of this study was to determine if degradation of residual crude oil could be enhanced with the use of soil amendments under the environmental conditions encountered in the Northern Great Plains. The objectives of this study were:

1. Test soil amendments which could be implemented by local landowners encumbered with oil contaminated soils.
2. Monitor the rate of decomposition of oil by measuring soil oil content at selected intervals.
3. Monitor soil physical and chemical parameters through time, including infiltration, soil temperature, and soil fertility.

LITERATURE REVIEW

Composition of Crude Oil

Petroleum hydrocarbons in crude oil can be broken down into three general classes: the saturate or aliphatic fraction, aromatics, and asphaltics (Atlas 1981). The saturate fraction includes alkanes (normal and iso), branched alkanes, and cycloalkanes. As a group, the saturate fraction accounts for the largest percentage of all compounds found in crude oil and are commonly referred to as waxes or paraffins. Alkanes with carbon numbers of up to C_{78} are known to occur, although most have carbon numbers from C_1 to C_{33} (Rowell 1977). The cycloalkanes (naphthenes) are saturated cyclic compounds isolated in the C_4 to C_{11} carbon range. Aromatic hydrocarbons contain a benzene ring and are found in both the low and high boiling range. The asphaltic fraction is composed of tar-like heavy fractions of crude, collectively known as resins. This fraction includes very complex ring structures, mixed condensed aromatic and cyclic ring compounds, and nonhydrocarbon polar components composed of nitrogen, sulfur, and oxygen (Bartha 1986).

Metals are also present in some crude oils, particularly vanadium (Baker 1970). Other metals including manganese, nickel, lead, selenium, mercury, zinc, chromium, and cobalt may also be present (Rowell 1977).

Effect of Oil on Soil

As oil is added to a soil it percolates downward through the profile, displacing air and water and adsorbing onto soil particles and organic matter. Heavy oil fractions are filtered out at the surface while lighter components pass to greater depths (Plice 1948). This wetting of the soil with oil brings about significant physical and chemical changes, most notably in soil redox potential, pH, temperature, wettability, and nitrogen and organic carbon content.

The redox potential of the soil changes from an aerobic condition to an anaerobic one if the oil content reaches high enough levels (Plice 1948; Schwendinger 1968; Rowell 1977). Oil increases the oxygen demand of a soil as it is oxidized during the degradation process (Dotson et al. 1970; Bartha 1986). Ellis and Adams (1961) reported a redox reduction from +0.833 mV to -0.982 mV in one soil following the addition of oil.

Changes in soil pH are dependent on the buffering capacity of the soil and the pH of the contaminating crude. Skujins and McDonald (1985) reported a pH of 2.7 for waste industrial oil. Dibble and Bartha (1979b) reported an increase of 0.3 pH units from 6.2 for an uncontaminated soil to 6.5 for soil contaminated with kerosene.

Soil temperature is primarily affected by the change in color of the soil surface. Most crude oils are brown, black, or green. These dark colors increase the absorption of radiant energy when spilled on a lighter colored soil. An increase of 5 to 9.5°C has been noted between oiled cultivated plots and unoiled, cropped plots at a depth of 15 cm (Toogood 1977).

Reduction in the wettability of the soil has been noted. Oil contamination over 0.5% by volume has been shown to reduce water uptake (de Jong 1980b). Severely contaminated sites exhibited considerably reduced soil-water uptake. Oil coated particles of soil resist the absorption of water, in one case reducing water uptake of a loamy soil by 50 to 75% during the first 5 minutes of wetting (Toogood 1977). After exposure to the atmosphere and sun, oil on the surface gradually thickens, eventually turning into a hard crust (Plice 1948; Mitchell, Loynachan, and McKendrick 1979). Once a surface crust develops, it becomes an effective barrier to water infiltration. Oiling of the soil also causes a breakdown of structure and dispersion of soil aggregates (Plice 1948). This may be due to the solvent properties of the lighter portions of the oil, which dissolve gums and waxes cementing aggregates together (Rowell 1977). Loss of structure and dispersion further affect soil-water relationships.

The carbon to nitrogen ratio (C:N) in an oiled soil rises dramatically due to the carbon contribution of the oil. Plice (1948) reported an organic matter content of 11.45% (≈ 6.7% organic carbon) and a total nitrogen level of 0.12% for a soil containing 6.77% oil. This translates to a C:N ratio of 56:1. Optimum C:N ratios for biological activity are in the 10:1 range (Donahue et al. 1983). Crude oils do contain 0.01% to 0.9% nitrogen (Toogood 1977; Brown et al. 1981) but this nitrogen is not necessarily available for use by organisms. Nitrogen does become available after degradation of the oil has been initiated.

Effect of Oil on Plant Growth, Germination, and Production

In the initial period following contamination, oil contacting vegetation adversely affects fine roots and root hairs (Bartha 1986). This inhibits water and nutrient uptake, killing some plants and suppressing the growth of others (Carr 1919; de Jong 1980b). Plants are able to take up some oil components from contaminated soil. In general, the smaller the hydrocarbon molecule, the more toxic the oil is to plants (Baker 1970). Volatile hydrocarbons have been shown to be extremely potent (Currier 1951 reported by Rowell 1977). Hydrocarbons within the 150 to 275°C boiling range (naphtha and kerosene) are also considered toxic and remain in the soil far longer than volatile fractions. Heavier oil fractions, however, have molecules that may be too large to penetrate plant tissues (Baker 1970), leaving them potentially less troublesome to plant growth.

Revegetation of an area contaminated with oil is governed by the adverse changes to the soil mentioned previously and direct effects of oil on plant growth and germination. These effects include the loss of a viable seed source, the creation of a poor seedbed, and an increase in competition for nutrients. Seed that was in the ground at the time of contamination is highly susceptible to oil. The more volatile oil fractions have a high penetrating power, entering through the seed coat and killing the germ (Plice 1948). In addition, the surface crust that develops after the oil has weathered is a poor seedbed and makes emergence difficult. Competition for available nutrients and oxygen also increases as the activity of decomposers is stimulated by the added carbon source (Gudin and Syratt 1975; Dibble and Bartha 1979a;

Mitchell et al. 1979). This makes it harder for plants to obtain the nutrients necessary for growth. Oil degradation may also produce toxic reduction products such as hydrogen sulfide gas which may inhibit or restrict plant growth (Bartha 1986).

Many investigators have examined the effects of oil on germination and production. Most of these effects have been negative although some beneficial results have been obtained on oiled soil. Murphy (1929) found that crude petroleum added at a rate of 0.17% by weight to soil delayed germination and reduced biomass by 36%. At a rate of 0.83%, biomass was reduced by 77%. Schwendinger (1968) conducted several laboratory experiments and reported germination of oat seeds was relatively unaffected in a loamy sand with up to 2.9% oil by weight. At 4.9%, however, germination was reduced by 73% when compared to the control. Dry weight biomass for ryegrass was reduced by 28% at an oiling rate of 2.7%. The growth of tomatoes, kale, and leaf lettuce was affected with 1.2% oil by weight in the soil. Uptake of water slowed considerably and the plants began to show typical nutrient deficiencies such as slowing of growth and yellowing of bottom leaves. In a greenhouse study conducted in Nigeria, Udo and Fayemi (1975) reported that germination was reduced 56% in soil oiled at a rate of 6.8% by weight. Plants were dehydrated and stunted. No seeds germinated in soil oiled at a rate of 10.6%. Germination was 100% at oiling rates of 1.1% but yields were only 70% of the control.

Mitchell et al. (1979) planted barley on field plots treated with 10 and 20 liters of oil per square meter. The first year, only 12% of the barley sprouted on the oiled plots versus the unoiled plots. Those that did sprout on the oiled plots grew only 5 to 10 cm tall compared to the 35 to 55 cm measured on the unoiled plots. The second year the unoiled plots had 5 to 7 times more above ground biomass than the oiled plots. By the end of the fourth year, the 10 liter plots had excellent growth while good growth was underway on the 20 liter plots. In a field study of yields of barley, oats, and wheat, de Jong (1980b) demonstrated that very small amounts of oil caused yield depression. Total above ground biomass was 70% of normal in soil contaminated with 0.05 to 0.25% oil and 43%, 17%, and 13%, for oil levels of 0.51 to 1.0, 2.01 to 4.0, and greater than 4.0%, respectively.

Plice (1948) compared yields of sorghum, cotton, soybeans, and field peas in soils oiled at 0.1, 0.5, and 1.0%. The average yield for the four crops was reduced to 86, 61, and 42%, respectively, of the control at the end of the first growing season. By the end of the third growing season, however, yields on the oiled plots had increased 5, 15, and 20%, respectively, over yields measured for the control plots. The increase in yield was attributed to an increase in nitrogen content on oiled plots from nitrogen in the oil and nitrogen fixation by microbes. Carr (1919) also noted the beneficial effects of oil on plant growth in a greenhouse study of unoiled and oiled soil. At an oiling rate of 0.75% by volume, biomass of soybeans more than doubled when compared to the unoiled pots. A linear relationship between oiling rate and yield was not found; a reduction in yield was noted in soil oiled at a rate of 1.75% when compared to the unoiled pots. At an oiling of 2.25%, no effect on biomass was recorded but biomass was reduced by 64% in soil oiled at a rate of 4% compared to unoiled soil.

Degradation of Crude Oil

As a pollutant, crude oil occupies an intermediate position between highly biodegradable substances and highly recalcitrant substances (Bartha 1986). The extent to which degradation of crude oil occurs and the rate at which the process proceeds is dependent on the type of oil, the nature of the contaminated soil, environmental conditions, and the nature of the microbial community (Dibble and Bartha 1979a; Atlas 1981).

Degradation is defined as the breakdown of petroleum components to compounds of lower molecular weight, or the transformation of petroleum compounds to more polar compounds of a carbon number equal to the parent compound (Atlas 1981). Degradation of oil yields carbon dioxide, water, and organic acids, along with smaller amounts of impurities, insoluble compounds, and microbial biomass (Francke and Clark 1974). High molecular weight, viscosity, and crystallinity are three properties of oil that inhibit biological oxidation and decomposition of oil (Dotson et al. 1970).

Pathways of Degradation

The four main pathways through which natural degradation occurs are evaporation, photo-oxidation, leaching, and biodegradation (Loynachan 1978). Evaporation and photo-oxidation may be minimized in soil systems because petroleum hydrocarbons are subject to rapid vertical infiltration. Bartha (1986) indicated that these two processes may be responsible for only 1 to 2% of the degradation of oil spilled on land. Leaching may also be of little importance in soil systems. Investigations into the leaching of oil sludges have demonstrated that leachates move only a limited distance in sandy loam and loam soils (Dibble and Bartha 1979b; Skujins and McDonald 1985). Biodegradation, then, remains as the dominant pathway for removal of crude oil contamination from soil.

The ability to biodegrade hydrocarbons is widely distributed among diverse microbial populations (Stone et al. 1942; Cansfield and Racz 1978; Atlas 1981). Over 100 species belonging to 30 genera of bacteria, yeasts, actinomycetes, and fungi present in soil can utilize hydrocarbons ranging from paraffin, kerosene, and gasoline, to asphalts, tars and rubber (Alexander 1961; Dotson et al. 1970; Jobson et al. 1974; Huddleston 1979; Atlas 1981). Microorganisms capable of degrading hydrocarbons are thought to be present in all prairie soils (de Jong 1980a) and are widely distributed in oil field soils, particularly in locations subject to frequent oil pollution (Francke and Clark 1974).

During biodegradation, microorganisms oxidize a portion of the carbon in crude oil for energy, and incorporate a portion into their cell wall (Loynachan 1978). Of the different hydrocarbon fractions present in crude, the straight chain alkane fraction is the most easily degraded by microbes (Stone et al. 1942; Schwendinger 1968; Rowell 1977; Atlas 1981). The rate of decomposition is decreased as the paraffin chain branches or forms ring structures (Schwendinger 1968). Cycloalkanes are particularly resistant to microbial attack and may be impossible to degrade, resulting in an end product of a tar-like residue (Atlas 1977; Atlas 1981). Higher molecular weight compounds are subject to attack although the rates are much reduced (Schwendinger 1968). Little is known about the degradation of the asphaltic and nitrogen, sulfur, and oxygen fractions. It has been shown, though, that asphaltics tend to increase during biodegradation (Bartha 1986). Dibble and Bartha (1979a) concluded that biodegradation of asphaltic compounds was dependent on the presence of saturated hydrocarbons.

Increasing the Rate of Biodegradation Using Soil Amendments

Early research regarding the removal of oil from soil concluded that natural processes proceeded at very slow rates and that little could be done to remedy the effects of oil unless the oil was completely removed (Knickman 1960 as reported by Schwendinger 1968). Continuing research has discounted this theory and shown that much can be done to enhance the biodegradation of oil in soil.

The process of biodegradation requires sufficient quantities of oxygen, nutrients, and water under favorable conditions of temperature and pH. The rate of biodegradation can be influenced through manipulation of the physical and chemical environment of the soil. Oxygen, nutrients and water can be provided through tillage, fertilization and irrigation to enhance the growth of microorganisms (Jobson et al. 1974; Gudin and Syratt 1975; Atlas 1977; Dibble and Bartha 1979a; Huddleston 1979; de Jong 1980b; Atlas 1981; Bartha 1986).

Tillage. The presence of free oxygen is essential to biodegradation. Frequent tillage of the soil has been suggested as a practical method to aerate the soil, although the depth of aeration is usually limited to the surface 15 cm by most farm equipment. Loynachan

(1978) monitored carbon evolution from tilled and non-tilled soils contaminated with Prudhoe Bay crude oil. He reported that more carbon was evolved from the disturbed soils than from the undisturbed soils and concluded that tillage should increase oxygen diffusion into the soil or increase hydrocarbon volatilization away from the soil. Jobson, Cook, and Westlake (1972) reported more rapid degradation of the saturate fraction of two crude oils under conditions of maximum aeration.

Mitchell et al. (1979) found that tillage improved infiltration into oiled plots and was effective in improving yields of barley. Plice (1948) found that tilled plots had naturally revegetated after five years while the non-tilled plots were still bare. Dibble and Bartha (1979b) used a subsoiler to aerate to a depth of 45 cm on an agricultural field contaminated with kerosene. After 21 months, the kerosene in the upper 30 cm had decreased from 0.87% to trace levels. Considerable quantities of oil remained in the 30 to 45 cm zone, however. They concluded that the main factor limiting biodegradation at this depth was reduced aeration.

Fertilization. Many investigators have reported beneficial effects of fertilization. Nitrogen and phosphorus are required in high concentrations for biodegradation to take place at higher rates (Atlas 1977). McGill (1977) developed a nomogram of nitrogen required for several types of oil in soil. The nomogram was developed by considering the amount and type of oil in the soil, and the amount of organic nitrogen available. Other nutrients including sulfur, iron, magnesium, calcium, and sodium are also required but in lesser amounts.

Sandvik, Lode and Pedersen (1986) reported fertilizer stimulated degradation of oily sludge applied to sandy soil. Fertilizer rates of 200, 400, and 600 kg N/ha showed oil reductions of 5, 18, and 22%, respectively, compared to the control in the first nine months and 20, 41, and 51% reduction after 32 months. Kincannon (1972) reported that the rate of biodegradation doubled with the addition of nitrogen and phosphorus. Jobson et al. (1974) found a significant increase in bacterial numbers after the application of nitrogen and phosphorus when compared to soils with no fertilizer. Raymond et al. (1976) found little stimulation of biodegradation by fertilizer in the first eight months after application. Fertilizer did not become a factor until 50% of the added oil was degraded.

Dibble and Bartha (1979a) reported 20 to 24% biodegradation of oil in fertilized soil columns after 120 days as compared to only 10% in the control. Evolution of carbon dioxide (CO_2) was highest for fertilizer rate of 60:1 C:N and a carbon to phosphorus (C:P) ratio of 200:1. Higher levels of fertilization inhibited biodegradation. This same result was also demonstrated by Brown et al. (1981) who measured CO_2 evolution from two different types of separator sludges added to soil. The waste from a petrochemical plant showed optimum degradation at a C:N ratio of 150:1 (adjusted from 350:1). When the C:N ratio was lowered to 20:1, degradation was inhibited. Waste sludge from a refinery had the highest rate of carbon dioxide evolution with the C:N ratio adjusted to 10:1. This rate of degradation was only slightly higher than the control, however, which had a C:N ratio of 110:1. McGill (1977) emphasized that excess nutrient additions can cause serious deleterious side effects from salinization, excess ammonia, and nitrate contamination of groundwater.

Irrigation. The availability of water is an important limiting factor controlling the rate of biodegradation in soil. Dibble and Bartha (1979c) indicated that oil sludge biodegradation was optimal at soil moisture contents of 30 to 90%. Skujins and McDonald (1985) concluded that, in an arid and semi-arid environment, moisture availability may be critical to biodegradation.

Temperature modification. The effects of temperature on biodegradation are not completely understood and conflicting results have been reported in the literature. Skujins, McDonald, and Knight (1983) found biodegradation took place only during periods of elevated temperatures when studying the landfarming of industrial waste oil. Francke and Clark (1974) noted a similar relationship in a study of the landfarming of waste oil and coolant. Biodegradation proceeded rapidly in warm weather (20 to 22°C) and slowed during cold rainy weather (5 to 15°C). Atlas (1981), on the other hand, reported that Colwell et al. (1978) found higher rates of biodegradation at 3°C than at 22°C in a study of oil contaminated beach sand with mixed microbial cultures. Other studies have shown the optimum temperature for biodegradation to be in the range of 18 to 30°C (Beerstecher 1954; Dibble and Bartha 1979a; Brown et al. 1981: Sandvik et al. 1986). Atlas (1981) concluded that temperature is not a limiting factor except as it affects the physical state of the oil and the availability of water (eg. frozen soils).

In order to modify temperatures in the field, several investigators have utilized plastic sheeting. Gudin and Syratt (1975) utilized black plastic sheeting to increase soil temperatures on soils contaminated with different types of petroleum hydrocarbons. They documented a higher rate of degradation on plots covered with sheeting than on plots amended with fertilizer and left uncovered.

Liming. The effects of pH on the rate of biodegradation have also been investigated under field and laboratory conditions. A soil pH of 7.0 to 9.0 has been suggested as ideal for both microbial growth and precipitation of any metals which may be associated with the oil (Dibble and Bartha 1979c; Huddleston 1979). Lime is commonly used to adjust soil pH where conditions warrant its use.

MATERIALS AND METHODS

Site Description

The Shay-Wait study site is located in the NW¼SW¼NE¼ of Section 24, T.35N., R.2W., Toole County, Montana, at an approximate elevation of 1067 m. The site is situated on rolling upland glaciated plains in north-central Montana (Figure 1). Dryland crop production, oil and gas production, and rangeland are primary land uses in the vicinity of the study area.

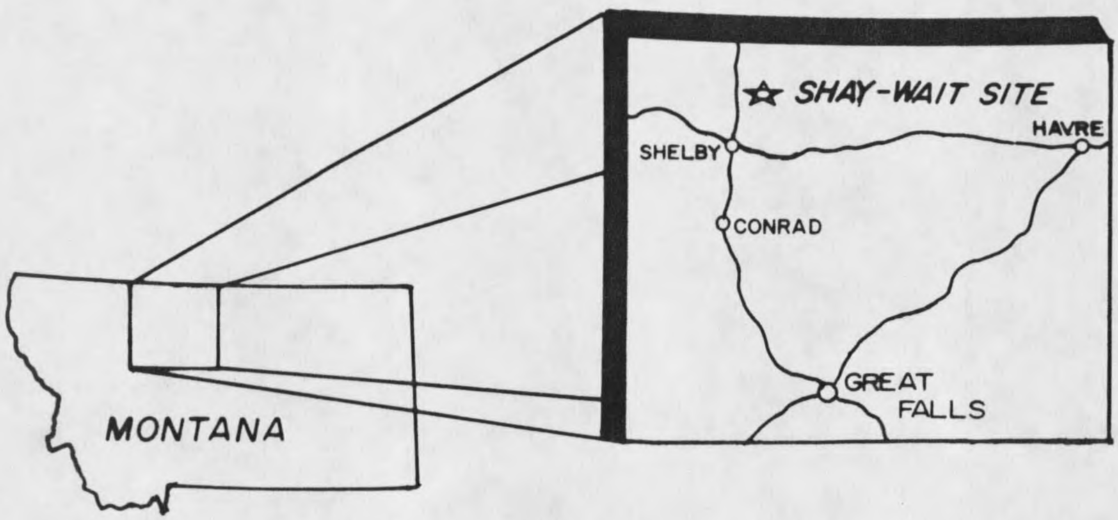

Figure 1. Location of Shay-Wait study site.

Precipitation of 25 to 35 cm per year in this area of the Northern Great Plains supports a shrub and grassland community composed predominantly of Artemisia cana (silver sagebrush), Atriplex spp. (saltbush), Opuntia polyacantha (prickly pear cactus), Artemisia frigida (fringed sagewort), Melilotus officinalis (yellow sweetclover),