THE INFLUENCE OF SEVERAL FACTORS ON THE BEHAVIOR

OF GRANULAR AND SPRAY FORMULATIONS

OF THREE HERBICIDES

By

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INTRODUCTION

Granular formulations of herbicides have been gaining popularity among farmers because of the several advantages they offer over the spray formulations.

Most of the more common herbicides are now available in either formulation, but little research has been done to determine the effectiveness of each formulation under a range of different environmental conditions.

Oklahoma is a state of environmental extremes, humid in the east and arid in the west. Performance of herbicides in Oklahoma has been erratic due to the environmental conditions which vary from year to year and reach extremes within short periods of time in any one season. Rainfall or lack of rainfall is one important environmental factor which determines the effectiveness of a herbicide in Oklahoma. In general, temperatures remain high throughout the summer and are not as variable as rainfall. However, the high temperatures attained in the summer are also an important factor in the performance of herbicides.

Due to the nature of the chemicals and their formulations, there is reason to believe that differences exist with reference to activation, persistence and effectiveness. The purpose of this study was to determine the performance of granular and spray formulations of three herbicides under a number of different environmental conditions.

REVIEW OF LITERATURE

Herbicides are applied either as sprays or in the granular form, each formulation having its own specific area of usefulness (12, 16, 29, 30, 53)¹. Formulation in this instance refers to the physical state of the inactive compound which serves as a carrier for the active ingredient and gives bulk to the herbicide. Spray formulations are applied as a liquid, while granular formulations are applied in a dry form, the active ingredient being impregnated on the carrier.

The nature of the carrier is very important in determining the effectiveness and the persistence of the herbicide (1, 14, 49, 50, 56, 62). The choice of carrier is limited by chemical compatability with the toxicant, physical relation with the toxicant and cost (45). As different carriers bind the toxicant with different degrees of tenacity with respect to volatilization and solubility, choice of the proper carrier can provide a desired period of persistence and a suitable rate of release (17). In formulating the toxicant, care must be taken to avoid any chemical reaction between toxicant and carrier that would impair the effectiveness of the toxicant. A desired combination is one which will enable the product to have a long shelf life and

¹Figure in parenthesis refers to literature cited.

to be effectively activated when applied to the soil (45).

Physical selectivity is one of the more outstanding features of granular formulations over sprays (4, 5, 15, 43, 60). When granular herbicides are applied to an emerged crop, the granules sift through the foliage of the plant down to the soil where they can act selectively. Sprays, on the other hand, are retained by the foliage and thus may not reach the soil. In some cases, leaf burn or other injury to the crop may result from retention of the herbicide on the leaves (15). Due to their physical selectivity, granulars sometimes fit better into the farming operation, since time of application with respect to the stage of growth of the crop is less critical: the crop may be emerged and application can be made without injury to the crop (36).

Superiority of one herbicide formulation over another appears to be determined to a large extent by environmental conditions, particularly the soil environment. Some reports show that, in some cases, spray formulations are more effective than granular formulations, while other reports state no difference or report better performance of the granulars (3, 10, 11, 27, 28, 42). Orsenigo (40) attributes better performance of spray formulations of amiben over the granular formulation at the same rate to lack of surface moisture at the time of application and to limited rainfall in the first week after treatment. Staniforth and Lovely (54) have reported that no differences of effectiveness were observed when granular and spray formulations of 2,4-D were subjected to various simulated rainfall conditions. In addition to

being as effective in controlling weeds as the spray formulation, Danielson (48) stated that the granular formulation was less likely to injure transplanted crops.

Under extreme drought conditions, EPTC in the liquid form at seven and one-half pounds per acre gave less than thirty days of crabgrass control, while the granular formulation at the same rate gave good control over a period exceeding thirty days (24, 31).

The main factors that affect effectiveness and persistence of a herbicide when applied to a soil are soil moisture and rainfall, organic matter and microbial population, soil structure and texture, chemical and photochemical decomposition, volatilization, temperature, and removal by plants (19, 22, 38, 59, 61). All these processes act simultaneously and show interaction effects (19). When soil moisture is at field capacity, microbial and chemical decomposition are favored. Excessive rainfall leads to greater loss by leaching, while insufficient rainfall results in greater loss by volatilization (44, 51). Loss of active toxicant also occurs due to fixation by soil mineral and organic colloids (13); fixation, however, reduces losses due to volatilization and leaching.

Soil Moisture and Rainfall

The herbicide must reach the seeds or seedlings being controlled in order to be effective. The soil solution provides a medium through which the toxicant is distributed through the soil and is brought to the site of action. Soil

moisture is also required to induce seed germination. Nongerminating seeds are generally not susceptible to herbicidal action. Burnside and Lipke (8) obtained results indicating that amiben was most effective when followed immediately by water application; as the rate of amiben per acre was increased, less water was required. Since spray formulations are distributed over the entire ground during application, less water is needed for their activation and distribution. Extra water is required with granular formulations in order to release the herbicide from the carrier and to distribute it throughout a given area. The more tenatiously the herbicide is held onto the carrier, the larger the granules, and the greater the percent active ingredient on the granule, the greater the amount of moisture required to distribute the toxicant (1).

Rainfall or overhead irrigation is the most effective source of moisture for activating and distributing herbicides in the soil (1, 32, 33, 34, 35). Better distribution and movement of the herbicide into the soil are obtained when the source of activating water is from above, since water movement occurs downward through the soil and distribution of the herbicide occurs partly by mass flow. The more insoluble a herbicide, the greater the importance of movement by mass flow. When the source of activating water is from below, such as in furrow irrigation, water movement occurs upward and there is a tendency to accumulate the toxicant at the surface of the soil.

Soil Microorganisms and Soil Organic Matter

The greatest loss of a herbicide in any soil occurs by microbial decomposition (2, 8, 9, 18, 20, 37, 39, 41, 46, 52, 58). All arable soils have such a vast and diverse population of microorganisms that almost any organic compound added to the soil is immediately acted upon (6). Any factor which favors the development of the soil microflora also favors the disappearance of herbicides from the soil. Gantz and Slife (20) have shown that CDAA and CDEC persist in the soil longer under conditions of low soil moisture and low temperature than under conditions of high soil moisture and high temperature. Roberts and Wilson (47) have obtained data indicating that the persistence of CIPC in the soil varies from three to twelve weeks depending upon the prevailing temperature and soil moisture. Similar results have been obtained by Burschel and Freed with amitrol and CIPC (9), Danielson et al (17), and Newman and De Rigo (37) with EPTC, Ogle and Warren with 2,4-D, TCA, IPC and NPA (39), and Switzer and Rauser (57) with simazine and atrazine.

Organic matter content is highly correlated with microbial population. Organic matter enhances the microbial population and microbial decomposition shortens the period of persistence of a herbicide in the soil.

Organic matter is also directly involved in decreasing the potency of an added herbicide through absorption of the herbicide (25). Sheets <u>et al</u> (52) reported that more herbicide is required in a high organic matter clay soil than in

a sandy soil of low organic matter in order to produce the same degree of weed control.

Leaching

Soil texture and structure affect persistence and effectiveness of a herbicide in an indirect fashion. In very sandy soils, losses due to leaching are of greater importance than losses due to microbial decomposition (44). In most sandy soils, the amount of organic matter is very low, indicating a small microbial population. Also, sandy soils lack the colloidal fractions which tend to reduce the magnitude of losses due to leaching. The amount and intensity of rainfall are very important factors in determining the extent of losses due to leaching. Too much rainfall produces leaching of the herbicides beyond the zone of effective control and may dilute the herbicide enough to render it impotent. The more intense the rainfall, the greater the loss by runoff and leaching.

Chemical and Photochemical Decomposition

The rate of microbial decomposition of herbicides generally follows a typical growth curve, with a lag period during which the microbial population adjusts to the new chemical introduced into the soil. This is then followed by a period of increasingly rapid rate of decomposition and finally a leveling off period at which the population reaches its optimum (9). Chemical and photochemical decomposition, on the other hand, begin almost immediately following application of a herbicide. In this case, the rate of decom-

position is linear and continues at the same rate until complete disappearance of the herbicide occurs. The losses through chemical decomposition occur mainly by hydrolysis and oxidation and are favored by some of the same factors that favor microbial decomposition (7). Photochemical decomposition occurs with some chemicals and is of special importance if a labile herbicide is applied to a dry, bare soil surface and bright, sunny days follow its application (19).

Volatilization

Herbicides which have high vapor pressures are most prone to losses by volatilization. Highly volatile compounds such as EPTC have a short period of persistence, while most compounds of low volatility show greater periods of persistence. In general, the greater the soil moisture content, the smaller the losses due to volatilization (21). This is due to the fact that when there is ample soil moisture much of the herbicide is in the liquid phase instead of the gaseous phase. In sandy soils, losses due to volatilization are greater than losses from high clay and/or high organic matter soils. Temperature is the most important single factor in determining losses due to volatility. The higher the temperature, the greater the loss by volatility.

<u>Removal by Plants</u>

Most plants take up a great number of compounds from the soil, including herbicides, with no apparent effect on the plant. Once the herbicide is removed by a plant from

the soil it is no longer potentially effective and, if absorbed in great amounts, its herbicidal effectiveness may be greatly reduced. Once a herbicide molecule has been absorbed by a plant, it can be decomposed within the plant or it can be rendered inactive by permanent binding.

It is evident that losses in effectiveness of a herbicide depend upon both the environment and the chemical and physical properties of the herbicide. Since there is little that can be done to control the environmental variables, attention has been focused on controlling the chemical and physical properties of the toxicant. Granulation of herbicides is one way in which physical properties of a herbicide can be regulated and its effectiveness and persistence controlled.

MATERIALS AND METHODS

The herbicides used in this study were NPA, amiben and dacthal. The commercial form of NPA used was the sodium salt of N-1-naphthylphthalamic acid in solution containing two pounds of active ingredient per gallon (sold as Alanap-3), and as a ten percent active ingredient granular formulation on 15-30 mesh attaclay.

Chemically, amiben is 3-amino 2,5-dichlorobenzoic acid and was used as a commercial formulation containing two pounds active ingredient per gallon. The granular formulation used was a ten percent active ingredient on 24-48 mesh attaclay.

Dacthal is the common name for the dimethyl ester of 2,3,5,6-tetrachloroterephthalic acid. It is available as a seventy-five percent active ingredient wettable powder and as a twenty percent active ingredient granular formulation.

All three herbicides are pre-emergence in action and have been shown to control a number of common weeds, including crabgrass and pigweed.

Greenhouse Studies

A Norge fine sandy loam soil of pH 5.5, organic matter content of 0.7 percent and a cation exchange capacity of 3.1 milliequivalents per one hundred grams of soil was used for all greenhouse studies.

Activation of Formulations by Overhead- and Sub-irrigation

Bioassay techniques (23, 41) were used to determine the effectiveness of the method of irrigation upon the activation of the two formulations of the three herbicides. The rates used were four and eight pounds of active ingredient per acre of NPA, two and four pounds per acre of amiben and eight and twelve pounds per acre of dacthal. Cimarron oats were used in the bioassay for NPA and amiben, and Redlan sorghum was used as a bioassay organism for dacthal. Soil was placed in metal flats eight by twelve inches in size and fifty seeds were planted in each pan. A total of four replications per treatment were made. The spray treatments were applied with a laboratory atomizer in fifty-two gallons of water per acre. The granular materials were sprinkled uniformly (using a shaker) over the area of treatment.

Overhead irrigation was simulated by applying the equivalent of one-half inch of rain in twenty minutes with a hand squeeze type sprinkler. This process was repeated for a total of one and one-half inches of rainfall.

Subirrigation was accomplished by setting the pans in water and allowing the water to be soaked up until the soil was wet to field capacity. A second subirrigation treatment was performed four days after the first watering.

Activation and effectiveness were determined by measuring the length of the coleoptile of the best ten seedlings per replication ten days after treatment. These measurements were expressed as a percent of coleoptile length as compared to the length of the check. A long coleoptile indicates slight herbicidal activity (i.e. 85-100 percent of the length of the check seedlings), while a short coleoptile (i.e. less than 85 percent) indicates that the herbicide was strongly activated. Decreasing coleoptile length indicates greater degree of activation. Each experiment was treated as a complete randomized block.

Persistence of Formulations at Different Soil Temperatures

Liquid and granular formulations of NPA, amiben, and dacthal were applied to soil and the soil was then held at temperatures of 60° , 75° , or 90° Farenheit. Persistence of each herbicide formulation was determined by bioassay technique, using Cimarron oats as a bioassay species for NPA and amiben and Redlan sorghum for dacthal. The rates in parts per million by weight of soil were NPA, 200 and 400; amiben, 10 and 20; and dacthal, 100 and 200. NPA experiments were assayed every two days, amiben experiments every ten days and dacthal experiments every twenty days.

Two experiments were conducted with NPA, amiben and dacthal in order to determine the persistence of each formulation at the three temperatures: (A) under conditions in which microbial decomposition and losses by volatilization were unrestricted; and (B) under conditions in which volatilization was held to a minimum. Also, an experiment was conducted with NPA in order to determine persistence under conditions of minimized volatility and minimized microbial activity. Each experiment was designed as a split split plot with four replications, the main split occurring between temperatures and the second split occurring between sampling dates.

Experiments in which microbial decomposition and volatilization losses were unrestricted were conducted in water baths in the greenhouse. Temperature variation was $\pm 3^{\circ}$ F.

Spray formulations were applied with a laboratory hand sprayer and the granular formulations were applied by hand. The herbicides were applied to evenly spread soil and then thoroughly mixed with the soil. The treated soil was placed in quart-size cans which were partially immersed in constant temperature water baths. Watering was performed daily with special care taken so as to maintain moist but not wet conditions at all times.

Representative vertical soil samples were collected periodically by means of a cork hole borer. Twenty-five seeds were planted in the collected samples and allowed to germinate and develop for five days in a seed germinator at alternating temperatures of 70-86 degrees F. Then the lengths of the coleoptiles were measured, and the values obtained were converted to percent of the length of the coleoptiles of the check treatment.

In the limited volatilization studies, volatility losses were minimized by placing the treated soil samples in airtight plastic boxes. Only initial watering was required. The samples were incubated in Mangelsdorf germinators for the 75° and 90° F. temperatures, and a constant temperature cabinet was used to incubate the samples maintained at 60° . Sampling and determination of persistence were performed as in the previous experiment.

Persistence of NPA formulations was determined under conditions of reduced microbial decomposition and volatilization. Volatilization losses were minimized by using sealed airtight containers and microbial decomposition was minimized by treating the soil with methyl bromide previous to applying the herbicide treatments. Twenty-four hours after the methyl bromide treatment, the treated soil samples were incubated at 60° , 75° , and 90° F. Individual airtight sealed boxes were used for each sampling date in order to avoid contamination by air exposure. Only initial watering was required. Determination of persistence was performed by the bioassay technique used previously.

Field Experiments

Experiments involving the comparison of the two formulations of each herbicide at two rates under prevailing environmental conditions were conducted at the Oklahoma Agricultural Experiment Stations near Perkins in 1962 and 1963, near Bixby in 1962 and 1963, and at Stillwater in 1963. The experiments near Perkins were planted with Hood soybeans in 1962 and with Clark soybeans in 1963. The Bixby tests were planted with Hill soybeans. The rows were spaced forty inches apart. The Stillwater plots were not planted with a crop. Spray formulations were applied with a tractor mounted sprayer. The granular formulations were applied with a hand applicator. Weed counts were taken in each experiment from within the twenty inch treatment band.

Daily temperatures and precipitation data for the different experiment locations are shown in Appendix Tables A and B, respectively. The most common weeds present were crabgrass (<u>Digitaria sanguinalis</u>) and pigweed (<u>Amaranthus</u> <u>retroflexus</u>).

Comparison of Formulations under Prevailing Environmental Conditions

Stillwater and Perkins:

The experiments conducted at Perkins in 1962 and 1963, and at Stillwater in 1963 were designed as a randomized complete block, with four replications at the Perkins location and eight replications at Stillwater.

At Perkins, the rates used in pounds of active ingredient per acre were: NPA, four and eight; amiben, two and four; and dacthal, eight and twelve. In 1963, the low rate of dacthal was six pounds per acre. The plots were two rows forty inches wide by twenty-five feet long. Weed counts were taken from an area of eight square feet per plot.

The experiment area at Stillwater was heavily seeded by broadcast with crabgrass (<u>Digitaria sanguinalis</u>), pigweed (<u>Amaranthus retroflexus</u>) and Italian ryegrass (<u>Lollium</u> <u>multiflorum</u>). The plots were one row ten feet long. Grass and broadleaf counts were taken from an area of four square feet per plot.

Bixby 1962, 1963:

The purpose of this experiment was twofold: to determine the effectiveness of the formulations of the three herbicides under prevailing climatic conditions and to determine the extent of weed control when applied at different crop stages and as pre-emergence treatment to the weeds. The soybeans were treated at three different stages of growth: immediately after planting, when the soybeans were one inch in height, and when the soybeans were six inches high.

Each experiment was designed as a split plot with four replications. The main plots were the different stages of application. The plots consisted of two rows twenty feet long. The rates of the chemicals used were: NPA, four and eight pounds; amiben, two and four pounds; and dacthal, eight and twelve pounds per acre. (In 1963, dacthal was used at rates of six and twelve pounds per acre.) The plots were band treated, twenty inches to the band. Previous to treatment, the plots were cultivated and hoed to insure that true pre-emergence effects would be observed. Weed counts for each plot were taken in four samplings of two square feet each.

Overhead and Furrow Irrigation in Activation of Granular and Spray Formulations; Stillwater, 1962

The experiment was designed as a split plot, with four replications, the split being between methods of irrigation. Each plot consisted of three ridges thirty inches wide by fifty feet long. The rates used, in active ingredient per acre, were NPA, four pounds; amiben, two pounds; and dacthal, eight pounds. Furrow irrigation was applied immediately after treatment and was continued until the ridges were completely wet. A second furrow irrigation was applied six days after treatment. Overhead irrigation was applied by sprinklers. Two inches of water were applied initially and one inch and two inches of water were applied three and seven days, respectively, after the date of initial irrigation.

The test was conducted during the month of August, during which only trace amounts of rainfall were recorded. Twelve days after treatment, grass and broadleaf counts were taken. Weed counts for each plot were taken in five samplings of two square feet each.

The most abundant weed species were crabgrass (<u>Digitaria</u> <u>sanguinalis</u>) and pigweed (<u>Amaranthus retroflexus</u>).

RESULTS AND DISCUSSION

Greenhouse Experiments

Activation of Formulations by Overhead- and Sub-irrigation

Overhead irrigation was more effective than subirrigation in activating both formulations of the three chemicals. However, all chemicals were activated to some degree by both methods of irrigation. Herbicidal activity (or activation) was slightly better for NPA than it was for amiben, regardless of method of irrigation.

The granular formulation of NPA was more effectively activated than the liquid under overhead irrigation at both rates. However, under subirrigation the low rate of the liquid formulation performed better than the granular formulation. The high rate of both formulations were activated to a similar degree by subirrigation (Table I).

The liquid formulation of amiben was activated more effectively than was the granular formulation under subirrigation (Table I). Both formulations were equally effective under overhead irrigation.

Amiben is quite soluble and overhead irrigation was sufficient to activate either formulation to about the same degree. The granular formulation of amiben at two pounds per acre was as effective as the liquid formulation at two pounds, and the granular formulation of amiben at four

TABLE I

ACTIVATION OF GRANULAR AND LIQUID FORMULATIONS OF THREE HERBICIDES UNDER OVERHEAD-AND SUB-IRRIGATION

Rate 1b/acre	Method of Irrigation	Formulation	Coleoptile Length as % of Check		
	N	IPA			
4 4 4 8 8 8 8	Overhead Overhead Sub Sub Overhead Overhead Sub Sub	Liquid Granular Liquid Granular Liquid Granular Liquid Granular	57.7 d 31.5 e 70.0 b 95.5 a 15.0 c 0.0 f 74.0 b 78.5 b		
	AMI	BEN			
2 2 2 2 4 4 4 4 4	Overhead Overhead Sub Sub Overhead Overhead Sub Sub	Liquid Granular Liquid Granular Liquid Granular Liquid Granular	61.0 e 61.0 e 86.5 b 93.5 a 46.5 f 47.3 f 67.3 d 78.0 c		
	DAC	THAL			
8 8 8 12 12 12 12 12	Overhead Overhead Sub Sub Overhead Overhead Sub Sub	Liquid Granular Liquid Granular Liquid Granular Liquid Granular	17.5 d 26.3 c 32.5 ab 33.0 a 10.7 e 15.3 d 31.0 ab 30.7 b		

¹Average of four replications. Within each herbicide, figures followed by the same letter are not significantly different; figures followed by different letters are significantly different (26, 55). pounds was as effective as the liquid formulation at four pounds.

As granular herbicides are formulated with a relatively large particle size (15-45 mesh), distribution on the surface of the soil is usually not as complete as would be true with a sprayed on formulation. However, distribution of the herbicide on the surface of the soil was not a factor in determining the degree of activation under overhead irrigation. The amount of water added was enough to dissolve and distribute the herbicide effectively.

Under subirrigation, the advantage of the liquid formulation over the granular formulation would appear to be due to a more even distribution of the herbicide on the surface of the soil during application and therefore to a more uniform movement into the soil.

In the overhead irrigated treatments, the liquid formulation of dacthal was activated better than was the granular (Table I). The low rate of the liquid formulation reduced coleoptile length to about the same degree as did the high rate of the granular formulation, indicating a requirement of a higher rate of the granular formulation to equal the performance of the liquid. The difference between formulation effectiveness with dacthal is probably due to the low solubility of the chemical itself. The liquid formulation is well distributed on the surface of the soil and offers more area for activation to take place.

Both liquid and granular formulations of dacthal were activated to the same degree under subirrigation.

Persistence of Formulations at Different Soil Temperatures

The slope of the curves at the three temperatures shown in Figures 1 and 2 indicates that under conditions of unrestricted microbial activity, persistence of NPA in the soil decreases as the soil temperature increases. This was found to be true for both granular and liquid formulations, for both high and low rates. With microbial decomposition restricted, temperature did not appear to have a material effect on the inactivation of the herbicide.

Figure 1 shows the persistence of two formulations and two rates, 200 and 400 parts per million by weight, of NPA under conditions of unrestricted volatilization and unrestricted microbial decomposition. At each temperature, the high rate of the granular formulation persisted longer than the high rate of the liquid. The low rate of the two formulations persisted equally with regard to time. When the soil temperature was maintained at 90° and 75° F., the visually observed effects of NPA disappeared completely after eight days. At 60° F., both rates of the liquid formulation and the low rate of the granular formulation disappeared between ten and twelve days after treatment. The high rate of the granular formulation persisted longer.

Figure 2 shows the persistence of NPA under conditions of restricted volatilization and unrestricted microbial decomposition. At 90° F. the high rate of the granular and liquid formulations persisted for twelve days, although the granular formulation retained its effective toxicity to a

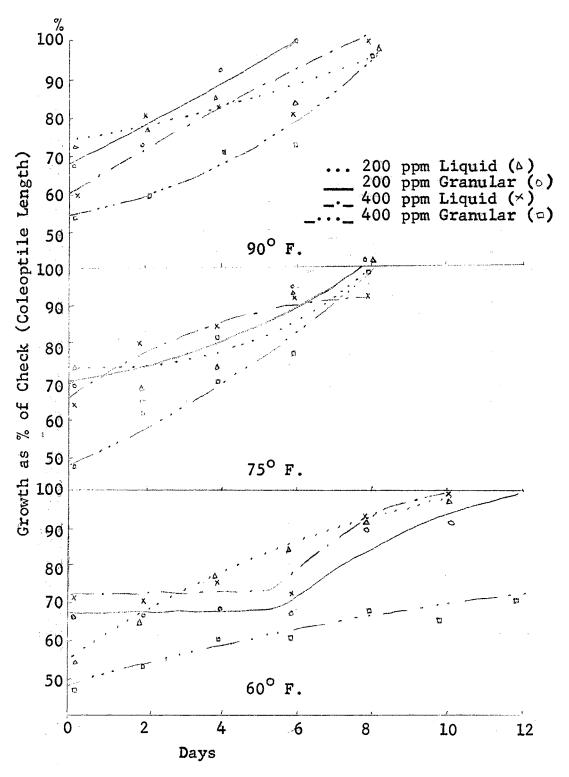
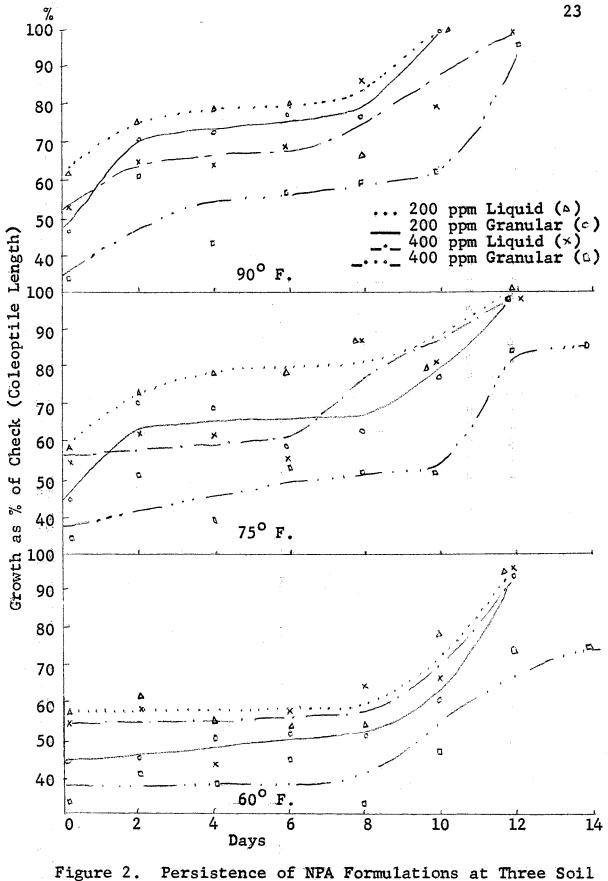


Figure 1. Persistence of NPA Formulations at Three Soil Temperatures under Conditions of Unrestricted Volatilization and Microbial Decomposition



Persistence of NPA Formulations at Three Soil Temperatures under Conditions of Restricted Volatilization and Unrestricted Microbial Decomposition.

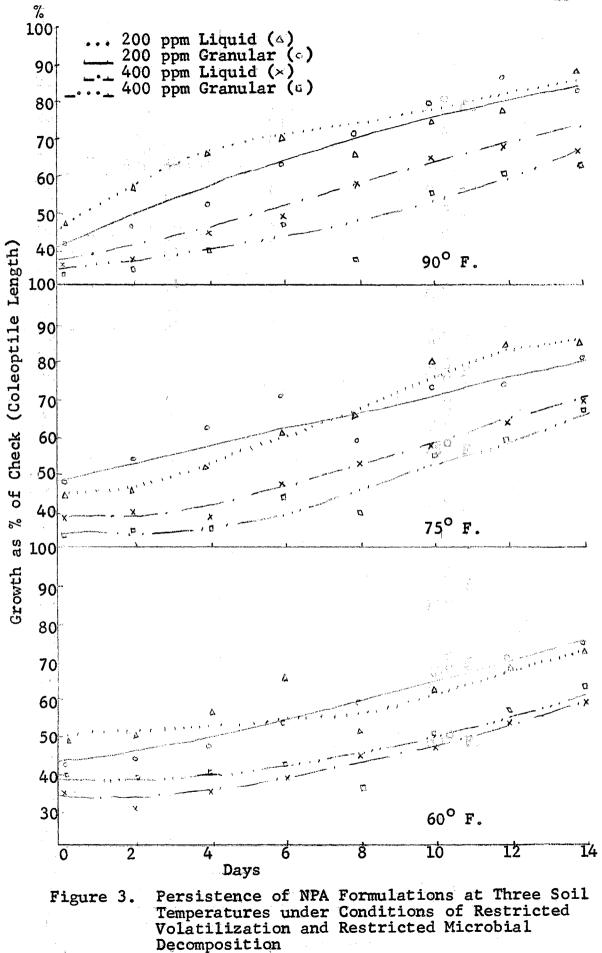
greater degree than did the liquid formulation. In other words, while both formulations were still present after ten days, the granular formulation inhibited growth of the bioassay species more than did the liquid formulation. The lower rates of the granular and liquid formulations had the same degree of toxicity and both formulations of the low rate disappeared by the tenth day after treatment.

At 75° and 60° F., the high rate of the granular formulation persisted beyond sixteen days, while the lower rates of both formulations, and the high rate of the liquid, were present in effective concentration only up to twelve days. The low rate of the granular and the high rate of the liquid formulation persisted equally as long at either 75° or 60° F.

Figure 3 shows the persistence of NPA formulations under conditions of restricted volatilization and restricted microbial decomposition. Disappearance of NPA under these conditions did not appear to be dependent upon the soil temperature.

Both liquid and granular formulations and both rates persisted beyond sixteen days at all three temperatures. Differences between formulations were not as sharp as those obtained in previous experiments. At all three temperatures, the activity of the lower rate was less than was the activity of the higher rate. The granular and liquid formulations were equally active at each rate.

Experimental evidence indicates that volatilization and microbial decomposition are very important factors in deter-



mining the persistence of NPA in the soil. High temperatures favor both types of losses and therefore it is expected that the herbicide will disappear from the soil faster when high temperatures prevail following herbicide application. When conditions were such that volatilization and microbial decomposition were unrestricted, NPA persisted only six to eight days. By restricting volatilization, the persistence of NPA was doubled; when both volatilization and microbial decomposition losses were restricted, the period of persistence tripled.

The fact that the high rate of the granular formulation persisted longer than did the high rate of the liquid formulation indicates that the granular carrier binds the toxicant and therefore reduces the amount of herbicide lost by volatilization. Binding by the carrier may also be a factor in determining the amount of moisture required to activate a herbicide under a given method of irrigation. When microbial decomposition and volatilization were both restricted, no difference between formulations was observed, indicating that the granular formulation is exposed to attack by soil microorganisms less than is the liquid formulation.

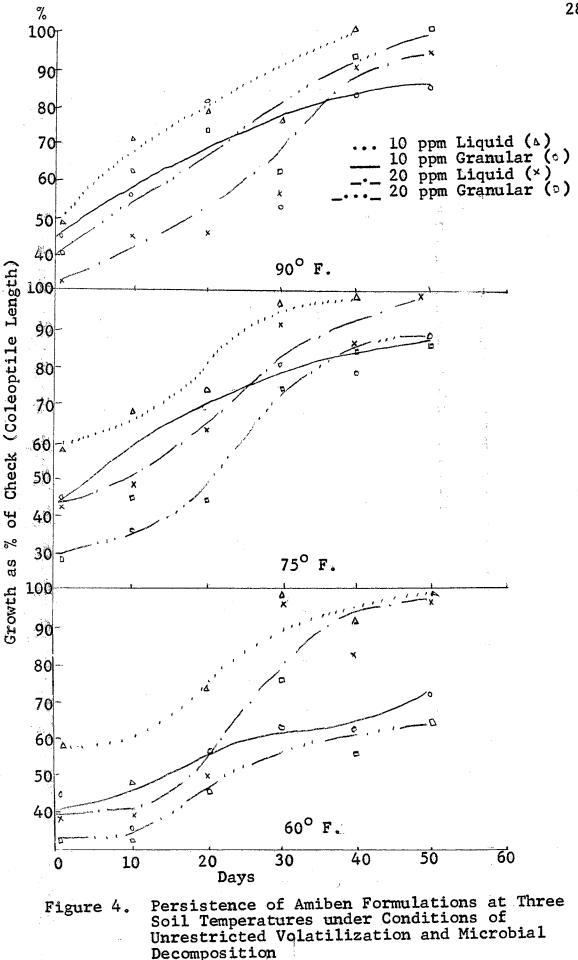
Erratic behavior of NPA under field conditions can be explained by the susceptibility of this herbicide to different environmental conditions. If hot weather follows application of the herbicide, its performance is likely to be poor due to its fast disappearance from the soil by volatilization and enhanced microbial decomposition. If rain

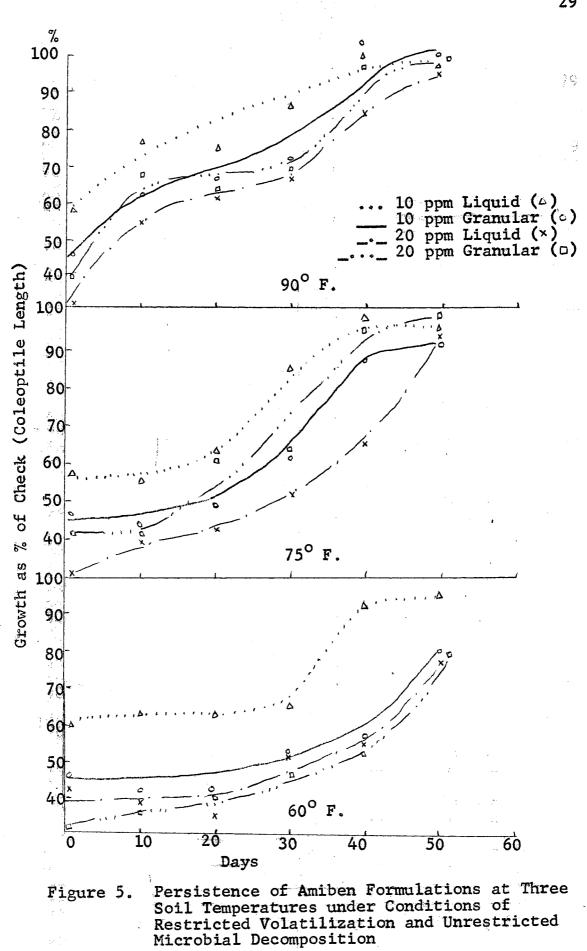
and low temperatures follow application of the herbicide, good weed control may be obtained. It is possible to obtain two to four days greater persistence by use of granular formulations if the temperature following application is high.

Figure 4 shows the persistence of the granular and liquid formulations of amiben, at 10 and 20 parts per million, under conditions of unrestricted volatilization and microbial decomposition.

At 90° and 75° F., the low rate of the liquid formulation persisted for thirty-five days. The other three treatments persisted at least fifty days. As the slopes of the curves in Figure 4 indicate, at 90° F. amiben disappears at about the same rate as it does at 75° F. At 60° F., the low rate of the granular and liquid formulations persisted for forty days, while the high rate of the granular and liquid formulations persisted over fifty days. Within the low rate and within the high rate, the liquid formulation persisted as long as the granular formulation.

Figure 5 shows the persistence of amiben formulations under conditions of restricted volatility and unrestricted microbial decomposition. The slopes of the curves again indicate that both formulations and rates of amiben persisted equally for fifty days at 90° and at 75° F. At 60° F. the low rate of the liquid formulation had disappeared by fortyfive days, while both rates of the granular formulation and the high rate of the liquid persisted longer than fifty-five days.





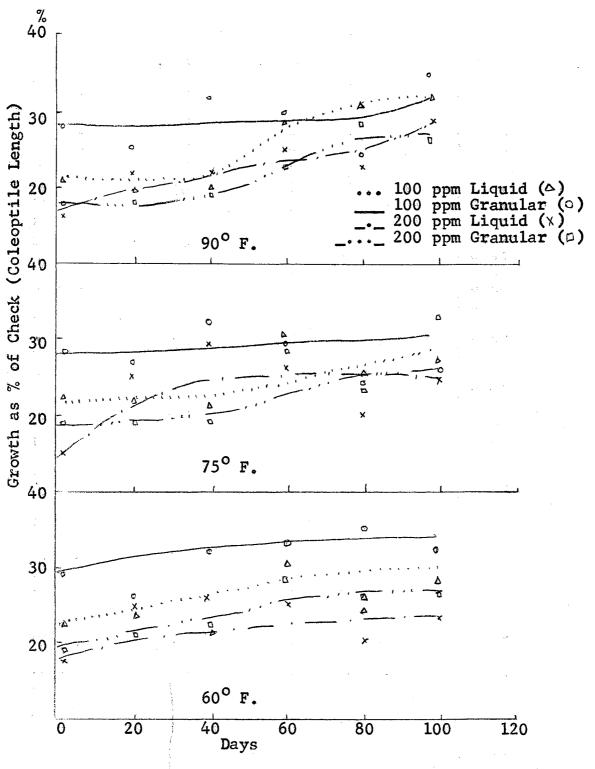
The granular formulation at each rate persisted the same length of time as the liquid formulation.

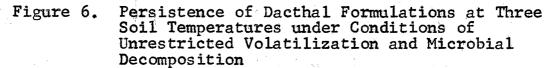
Experimental results show that losses by volatilization do not appear to be a very important factor in determining the persistence of amiben in the soil regardless of formulation. It appears that the main loss of amiben from the soil is probably by microbial decomposition.

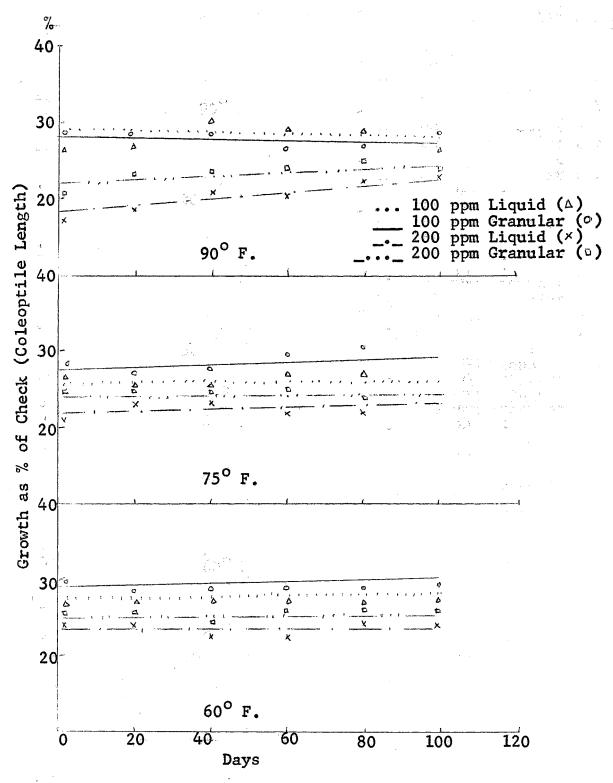
As shown in Figures 6 and 7, dacthal persisted over one hundred days in the soil at the three temperatures. Under conditions of unrestricted volatilization and unrestricted microbial decomposition dacthal tends to disappear slightly faster at 90° than at 75° F. However, the rate of disappearance is very low, indicating a long period of persistence and hence effective weed control. Persistence of this herbicide is probably due to its low susceptibility to microbial decomposition and to its very low volatility.

The granular and sprayed formulations persisted equally in the soil at each of the three temperatures. In dealing with a compound of low volatility and high resistance to microbial decomposition, the carrier is not as important as it appears to be with a chemical such as NPA which disappears faster from the soil when in the form of the liquid formulation than as a granular.

Under conditions of restricted volatilization and unrestricted microbial decomposition, temperature did not influence the persistence of dacthal. Each rate and each formulation persisted with its initial toxicity over one hundred days.







5 ¹ 1

Figure 7. Persistence of Dacthal Formulations at Three Soil Temperatures under Conditions of Restricted Volatilization and Unrestricted Microbial Decomposition

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The persistence curves shown in Figures 6 and 7 show that loss by volatilization does occur with dacthal. However, this loss is negligible over a period of one hundred days and does not appear to be a significant factor in the disappearance of dacthal from the soil at temperatures between 60° and 90° F. Since dacthal retains its initial toxicity when volatilization is restricted, it appears that microbial decomposition is negligible, at least during the first one hundred days.

Field Experiments

Activation of Formulations by Overhead- and Sub-irrigation

Conditions prevailing during the experiment were hot and dry. The daily temperatures exceeded 90° F. and only a trace amount of precipitation was recorded.

Table II shows the average number of grass and broadleaf weeds per ten square feet² of treated area. As was shown in the greenhouse, overhead irrigation was more effective than subirrigation in activating the herbicides.

Under overhead irrigation both formulations of NPA were equally effective in controlling grass and broadleaf weeds. Under furrow irrigation, however, the granular formulation gave better grass and broadleaf weed control than the liquid. Better performance of the granular formulation in the greenhouse and in the field was probably due either to reduced loss by volatilization from the granular formulation under high temperatures, or to loss by leaching following water

²Five two-square-foot random samples were taken per plot.

Chemical	Rate 1b/acre	Method of Irrigation	Formulation	Grass Counts	Broadleaf Counts
NPA	4	Overhead	Granular	1.3 d	3.3 e
NPA	4	Överhead	L iquid	3.0 cd	6.5 de
NPA	4	Furrow	Granular	7.8 cd	3.0 e
NPA	4	Furrow	Liquid	19.5 b	15.8 bc
Amiben	2	Overhead	Granular	1.8 d	6.3 de
Amiben	2	Ôverhead	Liquid	11.0 bc	14.5 bc
Amiben	2	Furrow	Granular	30.3 a	20.0 в
Amiben	2	Furrow	Liquid	41.3 a	37.3 a
Dacthal	8	Overhead	Granular	0.3 d	2.0 e
Dacthal	8	Överhead	Liquid	0.0 d	2.0 e
Dacthal	8	Furrow	Granular	1.0 d	7.5 de
Dactha1	8	Furrow	Liquid	7.3 cd	10.5 cd

TABLE II FURROW VERSUS OVERHEAD ACTIVATION OF TWO FORMULATIONS OF THREE HERBICIDES

¹Weed counts over an area of ten randomly chosen square foot areas per plot, average of four replications. Within each experiment figures followed by the same letter are not significantly different; figures followed by different letters are significantly different.

application.

The granular formulation of amiben gave better grass and broadleaf weed control than did the liquid formulation under overhead irrigation. Under furrow irrigation, the liquid and granular formulations were equally effective in the control of grass weeds, but the granular formulation was more effective in controlling broadleaf weeds.

In general, the dacthal formulations provided equally effective grass control under both types of irrigation. Under furrow irrigation, broadleaf weed control by the spray formulation was as effective as the granular but not as effective as either formulation in the overhead irrigated treatments. The liquid and granular formulations were equally effective in controlling broadleaf weeds under overhead irrigation. In the greenhouse tests, the liquid formulation of dacthal proved to be more effectively activated by overhead irrigation than was the granular formulation. Under field conditions, overhead irrigation activated both formulations to the same degree. However, in the greenhouse more precise control of moisture was possible. Subirrigation activated the formulations equally in the field and in the greenhouse.

Under field conditions moisture did not appear to be as critical as temperature for herbicidal effectiveness since ample moisture was provided. Better performance of the granular formulation appears to be due to the prevailing soil temperatures. The air temperature at time of herbicide application was 100° F. and the soil temperature at one inch below the surface was 99° F. At this temperature it appears that some of the herbicide applied as the liquid formulation would volatilize before reaching the surface of the soil and further volatilization would occur from the surface of the soil. The granular formulation prolongs the presence of the herbicide on the surface of the soil until sufficient moisture is provided for activation to take place.

<u>Comparison of Formulations under Prevailing Climatic</u> <u>Conditions</u>

Stillwater 1963:

Day temperatures prevailing on days following application of the herbicides were in the low seventies and high sixties. Night temperatures were in the forties and fifties. On the day of treatment, 0.24 inches of rain were recorded following application. During the next three days there was a total of 2.25 inches of rain.

Dacthal use resulted in excellent grass control, but broadleaf weed control was not as effective (Table III). Both rates and both formulations were equally effective in controlling grass weeds. The high rate of the liquid formulation was more effective than the low rate of the granular in controlling broadleaf weeds. The low rate of the granular formulation and the high rate of both formulations were equally effective.

Amiben was not as effective in controlling grass weeds as dacthal but broadleaf weed control by amiben was better.

TABLE III

FORMULATION EFFECTIVENESS UNDER PREVAILING CLIMATIC CONDITIONS: STILLWATER 19631

Chemical	Rate 1b/acre	Formulation	Number o Per Eight Grass	of Weeds t Square Feet ² Broadleaf
Dactha1	6	Liquid	6.0 b	22.0 b
Dactha1	6	Granular	4.6 b	16.6 bc
Dacthal	12	Liquid	0.8 b	2.0 c
Dacthal	12	Granular	6.0 b	14.5 bc
Check	0		74.0 a	65.0 a
Amiben	2	Liquid	18.0 b	1.2 b
Amiben	2	Granular	13.0 b	0.6 b
Amiben	4	Liquid	5.6 b	0.0 b
Amiben	4	Granular	1.6 b	0.0 b
Check	0		92.0 a	88.0 a
NPA	4	Liquid	42.0 b	27.2 bc
NPA	4	Granular	44.0 Ъ	34.0 b
NPA	8	Liquid	21.4 c	18.0 c
NPA	8	Granular	32.4 bc	21.2 bc
Check	0		88.0 a	70.0 a

¹Lines separate experiments. Within each experiment figures followed by the same letter are not significantly different; figures followed by different letters are significantly different.

²Average of eight replications. Sample area: eight square feet per plot.

Both rates and both formulations of amiben were equally effective in the control of grass and broadleaf weeds.

Grass and broadleaf weed control by NPA was not as effective as control by dacthal or amiben. The high rate of the liquid formulation of NPA gave better grass weed control than the low rate of both formulations. The high rate of the granular formulation was equally as effective as all rates of the liquid formulation. For broadleaf weed control, the high rate of both formulations of NPA was more effective than the low rate.

Poor performance of NPA appears to be due to the high solubility of the chemical and to the amount of rainfall recorded. Much of the chemical was leached out of the soil or washed away by runoff and its toxic effects reduced. The low solubility of dacthal prevented leaching of the chemical beyond the zone of weed seed germination and the rain was sufficient to activate the chemical effectively.

Perkins 1962 and 1963:

In 1962, between nine and eleven days after treatment there were 1.21 inches of rain. The prevailing day temperatures were in the high eighties and low nineties and the night temperatures were in the high sixties. In 1963, 0.5 inches of rainfall were recorded six days after treatment and five days later an additional 0.71 inches were recorded. The prevailing day temperatures following treatment were in the high eighties and low nineties.

Table IV shows the number of grass and broadleaf weeds per eight square feet in each plot.³ Weed control by all three chemicals was very good.

Both formulations and rates of NPA were equally effective for grass and broadleaf weed control in 1962 and in 1963. Grass control was slightly better in 1962 than in 1963.

The high rate of amiben controlled grass and broadleaf weeds better than the low rate did in 1962. In 1963, however, the low rate of the liquid formulation did not provide as good grass weed control as the low rate of the granular. The low rate of the granular gave equally good weed control as did the high rate of either formulation.

Dacthal formulations and rates gave excellent weed control in 1962 and 1963. There was no difference in weed control between rates or formulations.

Bixby 1962 and 1963:

Neither of the formulations of the three chemicals caused visible injury to the crop regardless of stage of crop development. Despite the extremely adverse climatic conditions encountered, all three chemicals gave good weed control.

Temperatures recorded after the first, second, and third treatments in 1962 were in the high eighties. Rainfall

³Average of four replications: samples per plot consisted of four two-square-foot samples.

TABLE IV

FORMULATION EFFECTIVENESS UNDER PREVAILING CLIMATIC CONDITIONS: PERKINS 1962 and 1963¹

Sec. 1			C	Weeds ²	Prese dia se	
Chemical	Rate 1b/acre	Formulation	Gra 1962	1963	Broadleaf 1962	
NPA	4	Granular	1.7 a	9.0 a	1.3 a	
NPA	4	Liquid	2.9 a	14.6 a	2.0 a	
NPA	8	Granular	1.7 a	6.6 a	0.8 a	
NPA	8	Liquid	2.0 a	13.6 a	0.8 a	
Amiben	2	Granular	6.4 ab	1.0 a	20.4 a	
Amiben	2	Liquid	7.3 a	16.6 b	22.2 a	
Amiben	4	Granular	0.8 c	1.0 a	5.7 Ъ	
Amiben	4	Liquid	1.7 bc	7.0 a	7.7 b	
Dacthal	8	Granular	0.8 a	ay an an an an	3.1 a	
Dactha1	8	Liquid	0.4 a		1.0 a	
Dacthal	6	Granular		2.0 a		
Dacthal	6	Liquid	80 80 60 60 60	1.0 a	67 68 69 69	
Dacthal	12	Granular	1.0 a	1.6 a	1.7 a	
Dacthal	12	Liquid	0.4 a	2.0 a	1.3 a	

¹Within each herbicide, figures followed by the same letter are not significantly different; figures followed by different letters are significantly different.

²Average of four replications. Sample area: eight square feet.

was recorded twelve days after the first application and amounted to 0.30 inches. Within the next five days rainfall totaled 1.27 inches. The second treatment was applied previous to the 1.27 inches. On June 2, 2.94 inches of rainfall were recorded and five days later an additional 3.71 inches were recorded. Two inches of rain fell during the following two days, bring about flooding conditions (i.e. standing water). The third treatment was applied two days previous to the 3.71 inch rain. The plots remained under water from four to seven days.

In general, the first NPA treatments were least effective in controlling weeds (Tables V and VI). The reason for this appears to be because of the high temperature following application and lack of moisture. These conditions are conducive to increase loss by volatilization. The high rate of granular NPA gave better weed control than the other rates for either formulation. This appears to show that there is less volatility from the granular formulation than there is from the liquid. This also was shown in the greenhouse studies. There was no difference between the second and third treatment dates for either grass or broadleaf weed control. The formulations were equally effective during the second and third treatments.

Amiben gave better grass weed control at the third treatment date than it did in the second and better in the second than it did in the first. The drought combined with the flood affected the first treatment more than the flood

TABLE V

FORMULATION EFFECTIVENESS UNDER PREVAILING ENVIRONMENTAL CONDITIONS: BIXBY 1962 AND 1963 - GRASS CONTROL

Chemica1	Rate 1b/acre	Formulation	5/12/62	5/23/62	Date of .6/5/62	Treatment .5/11/63	5/30/63	6/8/63	78.
NPA	4	Granular	16.0	8.0	9.1	11.0	5.0	6.0	- 18
NPA	4	Liquid	27.0	9.1	9.1	12.0	5.6	6.0	5.5
NPA	8	Granular	8.0	5.3	5.3	8.6	12.0	6.5	
NPA	8	Liquid	21.0	4.0	- 5.3	12.6	7.6	5.0	
Amiben	2	Granular	9.1	3.3	4.6	10.0	8.6	5.0	
Amiben	2	Liquid	10.7	15.3	4.6	7.0	11,0	6.0	
Amiben	4	Granular	8.0	1.3	2.7	7.0	4.6	7.0	
Amiben	4	Liquid	9.1	4.6	2.7	8.6	8.6	9.0	
Dacthal	6	Granular		·····	میں کا میں اور	5.6	3.6	1.0	
Dactha1	6	Liquid				6.0	3.6	1.0	
Dactha1	8	Granular	14.0	3.3	12.0	am ca es	629 Gto Can	c) == co	
Dactha1	8	Liquid	21.1	5.3	17.3				
Dactha1	12	Granular	12.7	10.7	9.1	2.0	2.6	5.0	1 L
Dacthal	12	Liquid	13.3	8.0	17.3	1.0	3.0	2.0	1.8.2.

¹Average of four replications: plants per eight square feet.

TABLE VI

FORMULATION EFFECTIVENESS UNDER PREVAILING ENVIRONMENTAL CONDITIONS BIXBY 1962 AND 1963 - BROADLEAF WEED CONTROL

Chemical	Rate 1b/acre	Formulation	5/12/62	5/23/62	Date of .6/5/62	Treatment 5/11/63	5/30/63	6/8/63
NPA	4	Granular	0.0	0.0	1.3	11.0	3.0	2.0
NPA	4	Liquid	8.0	0.7	2.7	14.0	7.0	2.0
NPA	8	Granular	2.7	1.3	5.3	7.6	6.0	0.6
NPA	8	Liquid	9.1	2.0	2.0	6.0	3.0	2.6
Amiben	2	Granular	5.3	0.8	1.3	2.6	1.6	1.6
Amiben	2	Liquid	6.7	4.0	2.0	7.6	0.6	3.0
Amiben	4	Granular	2.7	0.8	1.3	6.6	1.6	1.6
Amiben	4	Liquid	8.7	1.3	1.3	4.0	2.6	3.0
Dacthal	6	Granular				8.0	2.6	0.6
Dacthal	6	Liquid				7.0	3.0	1.6
Dacthal	8	Granular	8.7	4.8	4.7			
Dactha1	8	Liquid	10.7	1.3	7.3		i da ala	.) -
Dacthal	12	Granular	9.1	1.3	7.0	9.0	4.0	0.6
Dactha1	12	Liquid	2.7	1.2	4.7	1.0	3.0	0.6
		-						

alone affected the second and third treatments. The granular formulation of amiben gave better weed control than the liquid formulation at each rate in the second treatment date. In the other two dates the formulations were equally effective.

Dacthal formulations gave best weed control on the second date of application. Weed control due to the first and third treatment dates was the same. Both granular and sprayed on formulations were equally effective in controlling weeds.

In 1963, temperatures following the first treatment date were in the high eighties and 0.32 inches of rain fell on the day of treatment. Three days later, 0.25 inches were recorded and six days after treatment an additional 2.85 inches were recorded. The second application was followed by temperatures in the high seventies, then the eighties and nineties. Only trace amounts of rain were recorded during eighteen days following the second treatment. On the nineteenth day, 0.54 inches were recorded. The third application was made seven days previous to the 0.54 inch rain. Temperatures following the third application were in the high eighties and nineties.

In general, poorer weed control resulted from the first treatment than from the second or third treatment. Weed control was as good in the second treatment as it was in the third treatment.

The high rate of each chemical gave better weed control than did the low rate within each date of application. Formulations of NPA, of amiben and of dacthal were equally effective in controlling weeds in 1963.

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SUMMARY

Experiments were conducted in the greenhouse and in the field in order to determine the behavior of granular and liquid formulations of NPA, amiben and dacthal under various environmental conditions.

Both overhead- and sub-irrigation were effective in activating the three chemicals to some degree. Overhead irrigation was more effective than subirrigation in activating either formulation of the chemicals. In general, the liquid formulations were activated more readily than the granular when subirrigated under greenhouse conditions; while the granular formulations gave better weed control under field conditions. Better performance of the granular formulation in the field is related to less volatilization. Also, enough water was provided under field conditions to activate either formulation effectively.

Greenhouse results indicate that NPA has the shortest period of persistence in the soil at 60° , 75° and 90° F. and dacthal has the longest period of persistence in the soil. As the soil temperature goes up, persistence of NPA decreases due to enhanced microbial decomposition and volatility losses. The granular formulation of NPA appears to be slightly more persistent than the liquid formulation, but the formulations of amiben and dacthal appeared to be

equally persistent. Data collected showed NPA to be the most susceptible of the herbicides to loss by volatilization and microbial decomposition, while amiben was only moderately susceptible to losses by these factors. Dacthal gave only slight indication of being subject to volatilization, and its loss by microbial decomposition also appeared to be slow. Dacthal persisted over one hundred days, amiben for fifty and NPA for a maximum of twenty days at 60° F. under conditions of restricted volatilization.

The three chemicals provided good weed control at all field locations regardless of formulation. When hot dry weather followed herbicide treatments, the liquid formulation of the more volatile chemicals rendered poorer weed control than did the granular formulation. Heavy rainfall following application of the herbicides did not affect the efficiency of the herbicides to a great extent.

CONCLUSIONS

The granular formulation of NPA, amiben and dacthal is as effective as the liquid formulation for weed control, providing there is ample moisture for activation. If temperatures are high and the soil is dry, conditions are conducive to losses by volatilization, especially in the case of highly volatile materials such as NPA. Other substances such as dacthal have a low vapor pressure and hence undergo less loss by volatilization at high temperatures.

Under conditions of high temperatures and high soil moisture, conditions are favorable for losses due to microbial decomposition of the herbicide.

If sufficient moisture is provided for a good crop stand, the formulations are equally effective in controlling weeds.

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APPENDICES

APPENDIX A

Maximum and Minimum Daily Temperatures

	1962	196	1963					
Day	Aug. Max Min	April Max Min	May Max Min					
1 2 3 4 5 6 7 8 9 10	82 68 85 67 93 68 95 74 103 76		66 38 65 51 82 54 89 67 84 59					
6 7 8 9	$ \begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$		81 59 84 55 86 66					
L1 L2 L3	104 78 99 65 91 66		87 70 93 75 95 69 80 69 84 69 89 66 88 69 93 68 89 57 83 52 78 59 73 46 72 47					
L4 L5 L6 L7 L8	91 60 94 61 94 66 93 57 102 69	86 57 88 66 87 68	88 69 93 68 89 57 83 52					
L9 20 21	94 66 93 57 102 69 103 68 98 72 100 66 100 72 101 74	85 66 81 52 88 48 91 65	64 47					
22 23 24 25 26	97 68 84 59 88 50	* 70 46 66 47 66 48	63 46 75 51 87 68 86 55					
27 28 29 30 31	93 51 97 59 95 71 98 66 97 72	66 54 73 60 77 67 76 57 75 48	85 53 85 60 85 58 80 66 72 63					

Stillwater

*Date of treatment.

Maximum and Minimum Daily Temperatures

Day	June Max Min	1962 July Max Min	1963 June Max Min
$ \begin{array}{c} 1\\2\\3\\4\\5\\6\\7\\8\\9\\10\\11\\12\\13\\14\\15\\16\\17\\18\\19\\20\\21\\22\\23\\24\\25\\26\\27\\28\\29\\30\end{array} $	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	89 69 92 71 95 75 96 74 95 73 97 75 97 74 100 73 96 68 88 69 95 73 97 75 97 75 97 75 97 75 97 75 97 72 97 77 95 76 90 68 91 65	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$

Perkins

Maximum and Minimum Daily Temperatures

- 19-1.		1962	19	
Day	May Max Min	June Max Min	May Max Min	June Max Mir
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Day	19	62	xby 19		196 196	erkins 2	1963		illwater 19	63	
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APPENDIX B

Daily Precipitation (in inches)

*Date of treatment

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VITA

Juan Cardenas

Candidate for the Degree of

Master of Science

Thesis: THE INFLUENCE OF SEVERAL FACTORS ON THE BEHAVIOR OF GRANULAR AND SPRAY FORMULATIONS OF THREE HERBICIDES

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- Experience: Employed by Agronomy Department, Oklahoma State University 1959-1961 and by Mexican Department of Agriculture spring 1961. Graduate Research Assistant at Oklahoma State University 1961-1963.

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