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Effects of soil pH on the efficacy and persistence of several imidazolinone and sulfonylurea herbicides

Kermit Bruce Kirksey

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I am submitting herewith a thesis written by Kermit Bruce Kirksey entitled "Effects of soil pH on the efficacy and persistence of several imidazolinone and sulfonylurea herbicides." I have examined the final electronic copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Plant, Soil and Environmental Sciences.

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EFFECTS OF SOIL pH ON THE EFFICACY AND PERSISTENCE OF SEVERAL IMIDAZOLINONE AND SULFONYLUREA HERBICIDES

A Thesis
Presented for the
Master of Science
Degree
The University of Tennessee, Knoxville

Kermit Bruce Kirksey
May 1990

DEDICATION

To Mom, Dad, Kelly, Debbie, and the God who gave me each, I dedicate this thesis.

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ABSTRACT

Field and greenhouse experiments were conducted in 1988 and 1989 to determine the influence of soil pH on the persistence of imazaquin (2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1*H*-imidazol-2-yl]-3-quinolinecarboxylic acid), imazethapyr ±(2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1*H*-imidazol-2yl]-5-ethyl-3-pyridinecarboxylic acid), and chlorimuron (2-[[[[(4-chloro-6-methoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]benzoic acid). The concentrations were determined by using a standard response curve comparing the radicle length of corn 'Pioneer 3369A', treated with a known concentration of each herbicide, to the radicle length of corn from the treated areas in field samples.

Field studies were conducted at three locations in Tennessee representing West, Middle, and East Tennessee. The initial soil pH at all locations was 4.9 to 5.2. Soil pH was altered to 5.2, 5.5, 6.5, and 7.2 using hydrated lime ($Ca(OH)_2$). Herbicide treatments included imazaquin at 0.14 kg ai ha^{-1}, imazethapyr at 0.07 kg ai ha^{-1}, and chlorimuron-ethyl at 0.05 kg ai ha^{-1}. The experimental design used was a randomized complete block with a split-plot factorial arrangement of treatments, each replicated four times. The pH's were assigned to the main plots and herbicides were assigned to the split plots. All herbicides were incorporated using a field cultivator with S-tines followed by revolving baskets. Soybeans 'Asgrow 5474' were planted at all locations. Soil samples were taken 0, 8, 16, 32, 64, and 128 days after initial application. The soil samples were placed in a freezer and were used in the greenhouse portion of this experiment. The soil was allowed to air dry for 24 h, screened, and then placed in an acetate tube (15.24 cm in length). The tube was capped, placed in a tray filled with water and was allowed to sit for approximately one hour before planting pregerminated seed. Regression analysis was then performed for all herbicides at all locations with concentrations being regressed against time.

Results from the greenhouse bioassay indicate that soil pH significantly affected herbicide persistence. Imazaquin appears to be affected more by low soil pH than does imazethapyr. However, both herbicides dissipate quite rapidly from 0 to 32 days after application. There appears to be no significant difference in imazaquin persistence at pH 5.2 or 5.5. Imazethapyr appears to be more persistent when soil moisture is limited. Chlorimuron proved to be more persistent at pH 7.2 than at any other pH. Soil moisture conditions in 1989 were very high at all locations. Imazethapyr dissipated or moved out of the top 15 cm within 64 days. Crop injury was observed in 1989 with all herbicides. Rainfall occurred shortly after application and appears to have increased the amount of herbicide in the soil solution, making it more readily available for uptake by soybeans.

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CHAPTER I

INTRODUCTION

During the past several years, soil pH has become an increasing concern in crop production. For the majority of agronomic plants, the optimum soil pH for maximum plant growth is between 6.5 and 6.8 (69). The majority of soils in middle and east Tennessee have weathered from sedimentary bedrock, primarily limestone; however, metamorphic and igneous rocks are common parent materials in the mountainous part of the state (65). Limestone derived soils are usually slightly to moderately acidic. Loess soils predominate in west Tennessee and parts of middle Tennessee where most of the soybeans are grown.

The Soil Conservation Service reported in 1978 that west Tennessee leads the nation in soil loss due to sheet and rill erosion (8). In sheet erosion, the soil from an entire slope is removed in a uniform manner and rill erosion, which usually accompanies sheet erosion, is characterized by shallow gullies that are found primarily on bare soil (21). In 1988, the average soil loss in Tennessee exceeded 23 million tons; this is equivalent to 10.2 t ha^{-1} yr^{-1}. In some areas of West Tennessee, soil erosion exceeds 102 t ha^{-1} yr^{-1} (28). The available nutrients that are lost to erosion are relatively high because soil that is subject to erosion is higher in fertility than the lower subsoil.

In an erosion experiment conducted in Missouri, approximately 246.5 kg ha^{-1} of calcium and 97.5 kg ha^{-1} of magnesium were removed from the soil by erosion in continuously grown corn (*Zea mays* L.), whereas only 95.2 kg ha^{-1} of calcium and 32.5 kg ha^{-1} of magnesium were lost in a rotational system of corn, wheat (*Triticum aestivum* L.), and clover (*Trifolium spp.*) (69). There are a number of locations in Tennessee where the same crop(s) are grown year after year. Depending upon the cropping system, the soil is usually bare during the winter months following harvest. Since Tennessee ranks high in soil erosion, this contributes to the fact that most of Tennessee's soils are acidic.

Leaching, as a result of rainfall exceeding evapotranspiration, removes soluble salts, more readily available soil minerals, and bases (non-acidic cations such as Ca^{++}) (69). Soil pH is also lowered with the addition of nitrogen fertilizers, especially ammoniacal sources that produce H^+ during nitrification.

Farmers across the United States are leaning more toward a reduced-tillage or no-tillage cropping system as a means to control soil erosion (52, 62, 64). However, the response of farmers in Tennessee to no-till or reduced tillage is variable. Although Tennessee leads the nation in soil erosion, farmers still seem to be reluctant to adopt conservation programs. The hectarage of soybeans [*Glycine max* (L.) Merr.], corn, and grain sorghum (*Sorghum bicolor* L.) grown under a conservation plan declined from 1984 to 1988. Out of the 850,000 total ha that were planted in 1988, approximately 178,000 ha were grown under no-till or some means of reduced tillage. In other words, 80% of the total ha planted in 1988 utilized a conventional tillage system (68).

Farmers are becoming more aware of the importance of soil pH and are making an effort to adjust the soil pH to optimum conditions, regardless of the tillage system, for plant growth and they can do this inexpensively. Statistics show that the production cost of lime and/or gypsum to be approximately \$0.91 per planted ha of soybeans in 1988 and considerably less for corn (\$0.52) and cotton (*Gossypium hirsutum* L.) (\$0.32). The average cost of lime by the ton in 1989 was around \$9.00 per ton. Some local dealers are equipped with spreaders and can deliver and spread one ton of lime for \$6.00 per ha[1] (68).

With the movement toward conservation compliance in farming, the importance of chemical weed control will increase (28, 39, 41, 52, 54). By controlling weeds early with a preplant incorporated (PPI) herbicide, in conventional soybeans, or a preemergence (PRE) herbicide, in a conservation program with perhaps an early postemergence (POST) application, weeds should not present a problem as the soybean canopy is formed, depleting a weed's chance of survival.

Farmers who do not alter the soil pH to the optimum level may experience carryover problems with some of the newer preplant soybean herbicides. Two herbicide families that may present problems are the imidazolinones and sulfonylureas. The herbicides studied in this research were the imidazolinones imazaquin and imazethapyr, and chlorimuron, a sulfonylurea. Imazaquin and chlorimuron received marketing labels in the Spring of 1986 and imazethapyr was labelled in the Spring of 1989. During evaluation of these herbicides, some crop injury was observed (6, 9, 10, 22). Tennessee farmers observed significant crop injury in rotational crops following the use of these

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[1] Personal Communication. 1989. Knox County Farmers Cooperative.

herbicides. Residues of these herbicides from the previous year applications are believed to be the cause of the crop damage. Studies have shown that the imidazolinone family tends to be more persistent at low pH and the sulfonylurea family is more persistent at high pH (6, 9, 10). Crop injury appears to be associated with PPI methods rather than with POST application methods.

The primary objective of this research was to determine the soil pH at which these herbicides, applied preplant incorporated, cause carryover damage to rotational crops. By setting up a test with a range of pH's, it should be possible to determine the critical pH for degradation of each herbicide and thus avoid injury to rotational crops.

CHAPTER II

LITERATURE REVIEW

A. Review of Selected Herbicides

Chlorimuron

Chlorimuron (Classic™)is a highly effective sulfonylurea herbicide for control of economically important broadleaf weeds in soybeans. It can be applied PPI or PRE when mixed with metribuzin (4-amino-6-(1,1-dimethylethyl)-3-(methylthio)-1,2,4-triazin-5(4*H*)-one) and can also be applied postemergence (POST). Developed by E.I. duPont de Nemours & Co., Inc., chlorimuron bore the code name DPX-F6025. Chlorimuron is formulated as a dispersable granule and is also available in premixes with metribuzin (Canopy™, Preview™), and linuron (*N'*-(3,4-dichlorophenyl)-*N*-methoxy-*N*-methylurea) (Gemini™ and Lorox Plus™). The mode of action is blockage of biosynthesis of the essential amino acids valine and isoleucine by inhibiting the enzyme acetolactate synthase (also called acetohydroxyacid synthase). Metabolic inactivation by the soybean plant serves as the basis of selectivity. Chlorimuron is absorbed by the roots and foliage and is translocated apoplastically and symplastically. The acute oral LD_{50} of chlorimuron is greater than 5000 mg kg^{-1} of body weight (7, 9, 11, 22, 49, 55).

Imazaquin

Imazaquin (Scepter™) is a broad spectrum imidazolinone herbicide that controls many broadleaf weeds and some grasses. Imazaquin is currently being marketed by American Cyanamid Co. and was originally designated as AC-252,214. It can be applied preplant incorporated, preemergence, or postemergence. Imazaquin is available in premix formulations with chemicals such as pendimethalin (*N*-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine) (Squadron™); acifluorfen (5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoic acid), (Scepter O.T.™); trifluralin (2,6-dinitro-*N*,*N*-dipropyl-4-(trifluoromethyl)benzenamine), (Tri-Scept™); and alachlor (2-chloro-*N*-(2,6-diethylphenyl)-*N*-(methoxymethyl)acetamide), (Ala-Scept™). Imazaquin is absorbed by the roots and foliage of a plant and is rapidly translocated apoplastically and symplastically with accumulation in the meristematic tissue. Herbicide activity is due to the inhibition of the enzyme acetohydroxyacid synthase, which stops the synthesis of valine, leucine, and isoleucine, three essential amino acids. This causes disruption in protein synthesis which results in interference of DNA synthesis and rapid cessation of growth. Selectivity is due to differential metabolism and soybeans metabolize imazaquin rapidly to inactive forms. The oral LD_{50} of imazaquin is greater than 5000 mg kg^{-1} of body weight (5, 10, 23, 63).

Imazethapyr

Imazethapyr is a broad-spectrum systemic herbicide discovered and developed by American Cyanamid. The compound has provided excellent control of many major annual and perennial grasses and broadleaf weeds in soybeans. It is a member of the imidazolinone family. The trade name of imazethapyr is Pursuit™ and bore the code designation of AC-263,499. Imazethapyr is now available as a premix formulation with pendimethalin (Pursuit Plus™). Imazethapyr can be applied preplant incorporated, preemergence, or early postemergence. The mode of action is the same as that of imazaquin. Selectivity of imazethapyr is due to differential metabolism. Imazethapyr is absorbed by the roots and foliage and is translocated both apoplastically and symplastically. The oral LD_{50} of imazethapyr is greater that 5000 mg kg^{-1} of body weight (5, 6, 63).

B. Weed Control with Selected Herbicides

Chlorimuron

Chlorimuron is a preplant incorporated, preemergence, or postemergence herbicide developed for broadleaf weed control in soybeans. It has shown good to excellent control of several broadleaf weed species (9).

Perry et al. (53) investigated the toxicities of chlorimuron and imazaquin to corn in different soils in the South. The concentration range for chlorimuron was 0.16, 0.31, 0.63, 1.25, 2.50, and 5.00 ppm and the concentration range for imazaquin was 5, 10, 20, 40, 60, and 80 ppm, each being in many different soils from the South. Root length and root fresh weights were taken. Perry found there was no significant difference in the response of corn to the differing imazaquin concentrations but a difference was observed with differing chlorimuron concentrations. Chlorimuron was less toxic in the soils of the upper regions of the South including Georgia, Mississippi, Tennessee, and Texas, but was more toxic in soils of Florida and Louisiana.

Edmund and York (27) studied the factors affecting postemergence control of sicklepod (*Cassia obtusifolia* L. #[2] CASOB) using chlorimuron. Chlorimuron was applied postemergence in spray volumes of 50, 185, and 360 L ha^{-1}. They reported that good sicklepod control can be achieved with chlorimuron applied in spray volumes of 50 to 360 L ha^{-1}.

Gamble et al. (33) evaluated preemergence combinations of chlorimuron and imazaquin in a conservation tillage system. Chlorimuron was applied at 0.07 kg ai ha^{-1} and imazaquin at 0.28 kg ai ha^{-1}. Both provided excellent control of sicklepod.

Culbertson et al. (25) evaluated pitted morningglory (*Ipomoea lacunosa* L. # IPOLA) control with chlorimuron. Chlorimuron was applied postemergence at 9 g ha^{-1}. Pitted morningglory was controlled when chlorimuron was applied during the frist two weeks of growth.

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[2] Letters following this symbol are a WSSA-approved computer code from Composite List of Weeds, Weed Sci. 32, Suppl. 2. Available from WSSA, 309 West Clark Street, Champaign, IL 61820.

Decreased rates of chlorimuron and/or applications after two weeks resulted in significantly less control.

Reddy and Bendixen (56) evaluated soil-applied chlorimuron toxicity, absorption, and translocation on yellow (*Cyperus esculentus* L. # CYPES) and purple (*C. rotundus* L. # CYPRO) nutsedge. Soil applied chlorimuron (60 g ha^{-1}) decreased tuber sprouting by 80% in yellow nutsedge and by 30% in purple nutsedge. The decreased sprouting is probably due to the toxicity of chlorimuron to the tuber buds. Chlorimuron at 10 g ha^{-1} reduced shoot emergence by 99% in yellow nutsedge and 85% in purple nutsedge.

Adcock (1) studied the effect of preemergence herbicides followed by a postemergence application on sicklepod control. Applications of metribuzin followed by chlorimuron, chlorimuron plus metribuzin followed by chlorimuron, imazaquin followed by imazaquin, and imazethapyr followed by imazethapyr were evaluated for their herbicidal activities. All four programs reduced sicklepod numbers but adequate control was not achieved from any combination. Although sicklepod was not controlled, all programs reduced plant heights.

Allen and Banks (3) evaluated chlorimuron for broadleaf weed control in soybeans. Chlorimuron was applied PPI at 0.07 kg ai ha^{-1} and PRE at 0.035 kg ai ha^{-1}. The PPI treatments provided excellent control of tall morningglory (*Ipomoea purpurea* L. Roth # PHBPU), smooth pigweed (*Amaranthus hybridus* L. # AMACH), and prickly sida (*Sida spinosa* L. # SIDSP). There was some initial crop injury observed but no significant yield reduction occurred.

Imazaquin

Imazaquin is registered for use in soybeans as a preplant incorporated, preemergence, or a postemergence herbicide. Risley and Oliver (59) reported excellent control of common cocklebur (*Xanthium strumarium* L. # XANST), prickly sida, common ragweed (*Ambrosia artemisiifolia* L. # AMBEL) and pigweed (*Amaranthus spp.*) with imazaquin applied PPI, PRE, or POST at 0.14 kg ai ha^{-1} in combination with a grass herbicide. Although imazaquin has some activity on certain grass species, a grass herbicide is recommended for maximum grass control.

Aison and Harger (2) evaluated imazaquin for sicklepod control. Imazaquin was applied POST at 0.30 and 0.45 kg ai ha^{-1}. They observed good (86%) control of sicklepod. Imazaquin applied preemergence yielded 89% control of mexicanweed [*Caperonia castaniifolia* (L.) St. Hil. # CNPCA].

Umeda et al. (70) studied optimum timing of application of imazaquin for sicklepod and hemp sesbania [*Sesbania exaltata* (Raf.) Rydb. ex A. W. Hill # SEBEX] control. Imazaquin applied POST at 0.125 kg ai ha^{-1} gave optimal control of sicklepod when applied at the early cotyledon stage. However, higher rates (0.50 kg ai ha^{-1}) were required for 80% control of larger weeds. PRE applications (0.188 kg ai ha^{-1}) provided acceptable control of both sicklepod and hemp sesbania.

Retzinger et al. (58) evaluated imazaquin applied PPI, PRE, and POST at 0.14 kg ai ha^{-1}. Good to excellent (87 to 99%) control of prickly sida, pigweed, common cocklebur, and ivyleaf morningglory [*Ipomoea hederacea* (L.) Jacq. # IPOHE] was obtained with imazaquin applied PPI or PRE. Imazaquin applied POST resulted in lower control (63 to 94%).

Imazaquin, applied PRE, gave better control of burcucumber (*Sicyos angulatus* L. SIYAN) than when applied postemergence (20). However, satisfactory control was not achieved when compared to atrazine (6-chloro-*N*-ethyl-*N'*-(1-methylethyl)-1,3,5-triazine-2,4-diamine) applied preemergence.

Other studies (34) have been published regarding weed control with imazaquin indicating good to excellent control of Florida beggarweed [*Desmodium tortuosum* (S.W.) DC. # DEDTO], showy crotalaria (*Crotalaria spectablis* Roth # CVTSP), and coffee senna (*Senna occidentalis* L. # CASOC) by reducing the total root/shoot weights.

Minimum effective rates (MER) of imazaquin and chlorimuron were determined by Barrentine (14) on common cocklebur. MER is the quantity of herbicide that is required to provide at least 90% control. The mean observed MER's for imazaquin and chlorimuron for two-leaf cocklebur were 26.25 and 4.38 g ai ha^{-1}, respectively. MER's for 6-leaf cockleburs were 35.11 and 6.62 g ai ha^{-1}. Under moisture stress conditions and low relative humidity, chlorimuron at 8.75 g ai ha^{-1}, the highest rate, did not provide 90% control at the 6-leaf stage.

Imazethapyr

Imazethapyr is a relatively new herbicide that is labelled for soybeans and can be applied preplant incorporated, preemergence, or postemergence. It has shown excellent weed control possibilities in research conducted by American Cyanamid. Imazethapyr was labelled in the spring of 1989.

Imazethapyr was evaluated by Griffin et al. (36) from 1986 to 1988 to determine weed control possibilities of this new herbicide. Imazethapyr was applied postemergence at 0.05, 0.07, 0.09, and 0.11 kg ai ha^{-1} to soybeans and the results were compared to that of imazaquin (0.14 kg ai ha^{-1}), bentazon (3-(1-methylethyl)-(1*H*)-2,1,3-benzothiadiazin-4(3*H*)-one 2,2-dioxide) + acifluorfen (0.56 + 0.28 kg ai ha^{-1}), and fluazifop, ((+/-)-2-[4-[[5-(trifluoromethyl)-2-pyridinyl]oxy]phenoxy]propanoic acid) (0.22 kg ai ha^{-1}). Imazethapyr gave excellent control of common cocklebur when compared to imazaquin. Seedling johnsongrass [*Sorghum halepense* (L.) Pers. # SORHA] control was 91% when applied at rates of 0.07, 0.09, and 0.11 kg ai ha^{-1} at the 3 to 15 cm growth stage. Rhizome johnsongrass control was approximately 85%.

Imazethapyr has been shown to provide good to excellent control of ivyleaf morningglory, pitted morningglory, common cocklebur, redroot pigweed (*Amaranthus retroflexus* L. # AMARE), velvetleaf (*Abutilon theophrasti* Medic. # ABUTH), and jimsonweed (*Datura stramonium* L. # DATST) (32, 36, 38, 50).

Imazethapyr was granted an Experimental Use Permit in 1987 and in 1988 to treat 938 trials in several states of the South including Tennessee. Weed control was evaluated along with crop tolerance. All imazethapyr treatments were applied at 0.07 kg ai ha^{-1} PPI, PRE, and POST. Imazethapyr had excellent crop tolerance and exhibited 50% rhizome johnsongrass, 97% pitted morningglory, 82% entireleaf morningglory (*Ipomoea hederacea* var. integriuscula Gray # IPOHG) 83% ivyleaf morningglory, and 97% common cocklebur control as an average for all field trials (40, 67).

Studies by Boldt and Barrett (19) examined the use of naphthalic anhydride (NA) to reduce imazethapyr injury to corn. Two center rows of 4 row plots were treated with NA (1% w/w). Imazethapyr, at 0.071 or 0.140 kg ai ha^{-1}, was applied over the two center rows of each plot. Imazethapyr was applied PPI, PRE, and POST at three different stages of growth (spike, 4 to 5 leaf, 8 to 10 leaf). Optimum protection occurred when imazethapyr was applied PPI and at the spike stage. Corn was not safened when imazethapyr was applied POST at the 8 to 10 leaf stage.

C. Influence of Soil pH on Persistence of Selected Herbicides

During the past several years, soil pH has become an increasing concern in crop production. For the majority of soils, the optimum soil pH for maximum crop production is considered to be between 6.5 and 6.8 (69). Numerous studies have been conducted on the effects of soil acidity and alkalinity on plant growth. Soil acidity results in poor germination of plants, poor nitrogen and phosphorus uptake, sporadic growth, and poor yields (52). Soil acidity, neutrality, or alkalinity, is often considered to be the most important soil chemical property. Therefore, pH is one of the most frequently made soil tests. The pH of a particular soil may influence nutrient absorption and plant growth in one of two ways: 1) through the direct effect of the hydrogen ion; or

2) indirectly, through its influence on nutrient availability and the presence of toxic ions (21).

Soil pH influences the activity and adsorption of ionic herbicides by affecting the ionic character of the organic matter (O. M.) and clay colloids. The net negative charge of most soils is due to the abundance of negative charges on crystalline alumino-silicates and on organic matter. O. M. contains carboxylic acids and phenol groups that have a pH-dependent ionization with a pK_a of 5.2, which can determine the chemical character of a herbicide molecule and thus influence its adsorption on soil colloids. When the soil pH value is greater than the pK_a value, acidic herbicides exist predominantly in the anionic form and are repelled by negatively charged colloids.

Persistence Studies of Other Herbicides

Mersie and Foy (47) studied the adsorption and phytotoxicity of chlorsulfuron (2-chloro-N-[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl) amino]carbonyl]benzenesulfonamide) as affected by soil pH. Several soils varying in pH and other soil properties were used in this study. Chlorsulfuron adsorption decreased with increasing soil pH. Phytotoxicity to corn also increased with an increase in pH. A decrease in phytotoxicity occurred between pH 5.9 and 4.2. As the pH increased above 5.6, the negative charge on the soil colloids would repel the anionic form of the herbicide, thus increasing the chlorsulfuron availability for uptake by corn. These results agree with the studies performed by Frederickson (30) and Wehtje (71) on the effects of soil pH on chlorsulfuron activity.

Similar results (24) have been shown for other acidic herbicides such as 2,4-D ((2,4-dichlorophenoxy) acetic acid), dicamba (3,6-dichloro-2-methoxybenzoic acid), and chloramben (3-amino-2,5-dichlorobenzoic acid). Lavy (43) found that *s*-triazine herbicide availability for plant uptake decreased as the soil acidity increased.

Picloram (4-amino-3,5,6-trichloro-2-pyridinecarboxylic acid) adsorption has been found to be greatest in soils with low pH and high organic matter content (37).

Blumhorst et al. (18) studied the effect of soil pH on the mobility of cyanazine (2-[[4-chloro-6-(ethylamino)-1,3,5-triazin-2-yl]amino]-2-methylpropanenitrile). The major characteristic that distinguishes cyanazine from atrazine is the substitution of a propionitrile group for the isopropyl group on atrazine. This substitution increases the water solubility of cyanazine and decreases the basicity. Cyanazine therefore is less likely protonated in an acidic medium; thus it is more mobile than atrazine. Soil columns were treated with radiolabelled cyanazine and atrazine. The percent of radiolabelled herbicide leached was greater with cyanazine than with atrazine in the silty clay and silt loam soils. As the pH decreased from 6.8 to 4.7, the percentage ^{14}C-cyanazine recovered decreased, but the amount recovered was considerably more than for ^{14}C-atrazine. In soils of higher pH, greater mobility of both herbicides occurred as compared to the soils of lower pH's.

Imidazolinones

Imazaquin is widely used and imazethapyr should also be popular and potentially be used in soybean weed control. Since these two imidazolinones are relatively new, little research has been conducted on the persistence of these herbicides.

Imazaquin and imazethapyr are weakly acidic herbicides. They are predominantly uncharged molecules at low pH's and are anionic at high pH's. As the soil pH increases, increasing concentrations of weakly acidic herbicides should be available for root uptake, thus increasing phytotoxicity to sensitive plants. Examples of this type of behavior can be seen with dicamba and chlorsulfuron (24).

Imazethapyr. Imazethapyr is a relatively new herbicide for weed control in soybeans. Little information is available concerning the fate and influence of soil pH on the persistence, degradation, and availability of this herbicide. Several instances of carryover problems have occurred. Perhaps some of the research with imazaquin can provide some insight on imazethapyr availability and its fate.

In a study conducted by Loux and Slife (45), imazethapyr was applied to the soil and samples were taken each month for one year to determine the concentration of the herbicide at differing time intervals. Two soil series were used in this study, one being a Cisne silt loam (Mollic Albaqualf) and the other a Drummer silty clay loam (Typic Haplaquoll). Four months after initial application to the Cisne soil, there were no detectable signs of imazethapyr residue. Seventy percent of the chemical was lost during the first four months in the Drummer soil but residues were found during the rest of the sampling period.

In a separate study conducted by Flint et al. (29), greater than 90% of the imazethapyr recovered from soil columns was located in the top 8 cm in two soils. The two soils studied were a Pope silt loam (coarse-loamy, mixed, mesic, Fluventic Dystochrepts) and a Maury silt loam (fine, mixed, mesic, Typic Paleudalfs). Imazethapyr leaching was minimal in their tests.

O'Dell (51) studied imazethapyr transport in undisturbed soil columns. Water rapidly leached imazethapyr through the columns.

Imazaquin. Mills, et al. (48) studied the effects of tillage systems on the persistence of imazaquin and imazethapyr. The soybean tillage systems that were involved included conventional tillage (soil was moldboard plowed and disked), and no-tillage (wheat cover crop killed with paraquat before no-till planting). The results indicate that imazaquin was more persistent in the conventional tillage system than in the no-tillage system. Imazethapyr concentrations were not significantly affected by tillage systems. Corn injury was noticed the following year where imazaquin and imazethapyr had been applied to conventionally tilled soybeans. Injury was not observed where herbicides were applied to no-till soybeans the previous year (44, 46).

Basham et al. (15) evaluated imazaquin's persistence and mobility in three Arkansas soils in a two-year study. Imazaquin was applied PRE as high as 16X the recommended rate (0.14 kg ai ha^{-1}). Results show that under hot, dry field conditions, rapid dissipation of imazaquin occurred on a Taloka silt loam (Mollic Albaqualf) when no rainfall occurred within 2 weeks after application. In the second year, furrow irrigation was applied 7 days after initial herbicide application. Imazaquin phytotoxicity was much greater as dissipation was delayed. Imazaquin persistence was greater on the Sharkey silty clay (Vertic Haplaquept) than on the Taloka. Their results show that imazaquin is more likely to persist in soils with higher clay and/or O. M. contents and also with periods of dry weather. Persistence of fluometuron (N,N-dimethyl-N'-[3-(trifluoromethyl)phenyl]urea),trifluralin, and linuron has also been demonstrated in the Sharkey soil (60, 66).

Amin et al. (4) conducted an experiment to study the influence of wheat straw on soil reception of imazaquin. Wheat straw was applied at various densities, imazaquin was applied and the test was irrigated. Simulated rainfall moved imazaquin into the surface soil. In plots where no straw existed, imazaquin concentration decreased as the level of rainfall increased. In areas where reduced or no-tillage is employed, wheat straw reception of imazaquin may be the cause of soybean injury (32).

Field studies were conducted by Barnes and Lavy (13) to evaluate the activity of conventional and chemigation systems on the persistence and leachability of imazaquin and metolachlor (2-chloro-N-(2-ethyl-6-methylphenyl)-N-(2-methoxy-1-methylethyl)acetamide). Imazaquin and metolachlor were applied to a Taloka silt loam at 0.080 and 4.25 kg ai ha^{-1}, respectively. Soil samples were taken 1, 2, 3, 4, 6, and 8 weeks after application to depths of 1 to 8, 1 to 15, 15 to 22, 22 to 30, and

30 to 38 cm. Their results show that imazaquin adsorption increased as soil depth increased. Adsorption constants were highly correlated with decreasing soil pH and increasing clay contents. Metolachlor adsorption decreased with increasing depths, but was not affected by pH and clay changes. Application by chemigation increased the mobility of both herbicides with imazaquin accumulating primarily below 22 cm. Metolachlor did not leach below 22 cm. Imazaquin persistence was decreased when applied by chemigation. The reason for decreased persistence appears to be related to the large water volume associated with chemigation.

Renner et al. (57) examined the influence of rate, method of application, and tillage on imazaquin persistence. Imazaquin was applied PPI and PRE at 35, 70, 140, and 280 g ai ha^{-1}. Corn was planted over the entire test and soil cores were taken at different intervals for analysis. Imazaquin applied PPI at 280 g ai ha^{-1} persisted longer than when applied PRE. When corn was planted the second year into the previous year's imazaquin application, significant injury did occur. In agreement with other research, more injury was observed with the PPI treatments than with PRE treatments.

Goetz et al. (35) conducted an experiment to determine the soil solution and mobility characteristics of imazaquin. The study was conducted on five different soils ranging from clay to sandy loam. Their results suggest that imazaquin mobility is dependent upon soil type. Imazaquin was more mobile (R_f=0.90) in the Sumter clay (Rendollic Eutrochrepts) and in the Decatur silt loam (Rhodic Paleudults), than in any other soil, even though the Sumter soil was higher in clay and organic matter. Imazaquin was, however, less mobile in the clay soil with the lower pH value. Sorption was enhanced by lowering the pH in all soils. Results indicate that soil sorption is governed by the pH-dependent charged surfaces from aluminum and iron oxyhydroxides and kaolinite.

Kendig et al. (42) evaluated the response of winter wheat to carryover from imazaquin, chlorimuron, imazethapyr, atrazine, fluometuron, norflurazon (4-chloro-5-(methylamino)-2-(3-(trifluoromethyl)phenyl)-3(2H)-pyridazinone), trifluralin, clomazone (2-[(2-chlorophenyl)methyl]-4,4-dimethyl-3-isoxazolidinone), metolachlor, and alachlor, all soil applied. Wheat was planted into plots where the herbicides had been evaluated on cotton, grain sorghum, and soybeans, at nine different locations. Trifluralin, chlorimuron, alachlor, metolachlor, and the imazethapyr plots did not show any yield reduction. In wheat following soybeans, clomazone caused injury (30 to 60%) to wheat at most locations. Imazaquin from two years' use (0.14 kg ai ha^{-1}), gave 78% injury to wheat when evaluated on a Sharkey silty clay soil. Imazaquin was also applied at the time of wheat planting and caused a 50% reduction in yield.

Sulfonylureas

The sulfonylurea herbicides represent a major advance in weed control technology. Important factors that contribute to the rapid success of the sulfonylureas include greater selectivity, low dosage, environmental compatibility, and groundwater safety. With more research being conducted in this family, advances will be made that could have a significant impact on weed control (16).

Chlorimuron. In a study conducted by Bloodworth and Shaw (17), crop injury was observed when using sulfonylurea herbicides. The injury occurred when using chlorimuron plus linuron and chlorimuron plus metribuzin in a wheat-soybean double cropping system. The soil pH of the experimental test area was 7.8, which exceeds the maximum designated by the herbicide label. Crop injury symptoms typical of these two chemical formulations indicate that chlorimuron was the primary component causing soybean injury. Farmers in the Southeast who practice this double cropping system may experience this type of injury. Therefore, soil pH is a major factor to consider when using the sulfonylurea herbicides.

Schmitz et al. (61) studied chlorimuron persistence. Different rates of chlorimuron were applied preemergence to soils of pH 5.3 and 7.3. Two phases of chlorimuron degradation were discovered. The early phase, soil samples taken from 0 to 32 days after application, showed rapid degradation of chlorimuron at all soil pH levels. Soil tests taken from 32 to 128 days past treatment revealed decreased chlorimuron degradation rates in all soil pH levels. They conclude that chlorimuron is more persistent in soils with a high pH. Therefore, there is a higher risk of crop injury to a subsequent crop.

Injury to soybeans was observed by Fuqua (31) in no-tillage treatments using chlorimuron + metribuzin and chlorimuron + linuron. Soybean injury appeared to be related to excessive moisture levels in the crop residue. With an increase in the amount of residue, there is an increase in the amount of crop injury.

Research was conducted from 1985 to 1987 to evaluate weed control of imazaquin and chlorimuron (12). In 1988, cotton was planted into the 4 chlorimuron and 8 imazaquin treatments. Cotton injury was observed early in the growing season in all treatments, but cotton stand was not significantly different from the untreated plots. By 4 weeks after planting, cotton height from 3 of the 4 chlorimuron and 7 of the 8 imazaquin treatments were significantly reduced. Chlorimuron reduced seedling weight but not as much as imazaquin. Yield of 1988 cotton was significantly reduced where imazaquin (0.14 kg ai ha^{-1}) was applied PPI and POST during 1985-1987.

CHAPTER III

EFFECTS OF SOIL pH ON THE EFFICACY AND PERSISTENCE OF IMIDAZOLINONE AND SULFONYLUREA HERBICIDES

A. Introduction

Soil pH is now recognized as one of the most important factors that is necessary for maximum plant growth. More farmers in the Southeast are making an effort to alter their soil pH to the optimum level. However, there are still areas in Tennessee where soil pH is below desired levels. The addition of nitrogen fertilizers decreases the soil pH. Excessive rainfall causes leaching of calcium and magnesium to occur thus lowering the soil pH (69).

The introduction of low dosage herbicides has already had an impact on weed control technology. The imidazolinone and sulfonylurea classes of chemistry are two new families that were introduced in the early 1980's that have low dosage weed control capability. Recent research, especially with imazaquin, imazethapyr, and chlorimuron, indicate that soil pH influences carryover potential. Applied preplant incorporated, these herbicides may have carryover potential to other rotational crops. The primary objective of this research was to determine the soil pH at which imazaquin, chlorimuron, and imazethapyr, applied pre-plant incorporated, cause carryover damage to rotational crops. It should be possible to determine the critical pH's for degradation of these herbicides.

B. Materials and Methods

Field Research

Field studies were conducted in 1988 and 1989 at three locations in Tennessee: Plant Sciences Field Laboratory, Knoxville; Milan Experiment Station, Milan; and Highland Rim Experiment Station, Springfield. These studies were performed to determine the effects of different pH levels on the persistence and carryover of imazaquin, imazethapyr, and chlorimuron.

The soil at Knoxville was a Melville silt loam (clayey over loamy-skeletal, mixed Agric Cryoborolls) with 0.81% O.M. and an initial pH of 5.2. The soil at Milan was a Calloway silt loam (fine-silty, mixed, thermic Glossaquic Fragiudalf) with 0.56% O.M. and an initial pH of 4.9. At the Springfield location, the soil was a Dickson silt loam (fine-silty, siliceous, thermic Glossic Fragiudults) with 0.56% O.M. and an initial pH of 5.1. Each experiment was established on soils of low pH in order to ease adjustment of pH.

Experimental design used at all three locations was a split-plot factorial arrangement of treatments in a randomized complete block design with each treatment replicated four times. pH treatments were assigned to the main plots and herbicide treatments were assigned to the split plots.

Soil samples were taken in the winter of 1988 at a depth of 12 to 15 cm and a soil titration was conducted to construct a response curve that was used to determine the lime requirement for each main plot.

The pH's that were obtained after liming were 5.2, 5.5, 6.5, and 7.2. These main plot treatments were replicated four times. Hydrated lime ($Ca(OH)_2$) was used and was applied by hand to the desired plots in March and April of 1988 and in May and June of 1989. Soil samples were taken at each location to determine soil pH at approximately four weeks prior to planting and at planting. Soil samples were taken throughout the growing season and at harvest to monitor soil pH levels.

A conventional tillage system was implemented at each location during both years. Before planting of soybeans, the experimental area at each location was disked and herbicides were applied PPI. The herbicide treatments were the same both years at all locations. The treatments included chlorimuron (0.05 kg ai ha^{-1}), imazaquin (0.14 kg ai ha^{-1}), imazethapyr (0.07 kg ai ha^{-1}) and an untreated control. Plots were maintained weed free with acifluorfen + bentazon (0.187 + 0.374 kg ai ha^{-1}), (applied as Storm™) for broadleaf weed control, and with sethoxydim (1.75 L ha^{-1}) for grass control, both applied at two week intervals if needed. When the control plot needed to be sprayed the entire test was sprayed in order to ease the soil sampling task.

'Asgrow 5474' soybeans, a determinant, medium maturing cultivar, were planted June 1, 1988 and July 28, 1989 at Knoxville; June 7, 1988 and July 5, 1989 at Springfield; and July 6, 1988 and June 2, 1989 at Milan. Planting of soybeans at Springfield and Knoxville in 1989 was delayed because of high soil moisture content. Soybeans were planted in 8-row plots with a row spacing of 76.2 cm and 7.6 m in length at all locations except Milan where the row length was 9.1 m. The eight-row plots were divided into two four-row plots that were used for different years of chemical applications. Herbicide applications were applied with a CO_2 pressurized backpack sprayer equipped with a four nozzle boom with 8002 flat fan tips having a nozzle spacing of 51 cm. A water carrier volume of 238 L ha^{-1} and a pressure of 276 kPa was used in 1988. A water carrier volume of 255 L ha^{-1} and a pressure of 276 kPa was used in 1989. After all chemical treatments were applied, the entire experimental area was incorporated with a field cultivator equipped with S-tines followed by rolling baskets.

Soil samples were taken with a soil probe to a depth of 15 cm immediately after planting soybeans. The soil was placed in a Whirl-Pak™ bag (0.53 L) and then frozen. Soils were sampled 0, 8, 16, 32, 64, and 128 days after herbicide application. Samples were used in the greenhouse portion of this research. Soil samples were pulled from the two center rows to help minimize contamination from adjacent plots.

Soybean yields were obtained at all locations, both years with the exception of Springfield and Knoxville in 1989. Wheat was no-tilled into the soybean stubble in order to minimize movement of the chemical during the winter. Wheat was planted on December 2, 1988 at Knoxville and December 5, 1988 at Milan. Wheat was not planted at Springfield due to late soybean harvest. Wheat yields were not obtained at either location.

Carryover effects were measured in 1989 at the Milan location by measuring plant heights 16 and 32 DAT. The inhibition of soybean elongation was determined by comparing the heights of the soybeans in the treated plots with the heights of the control plots within the same main treatment.

Bioassay

The greenhouse study was initiated in 1989 at the Plant and Soil Science greenhouse located on the Agricultural Campus of the University of Tennessee at Knoxville. Soil samples collected from the first and second year of this research were used in a corn 'Dekalb 3369A' root bioassay to determine the amount of chemical left in the soil over time. This variety has been shown to be susceptible to the imidazolinone herbicides imazaquin and imazethapyr.

The environmental conditions of the greenhouse were below that which would have been desired. Daily temperatures averaged in excess of 40 C. The greenhouse is quite old and air circulation was near impossible. All of the windows were opened and shade cloth was placed over the benches used for this experiment.

Corn seeds were placed in trays layered with wet paper towels. The seeds were placed in the tray with the endosperm side down for quicker germination. The trays were then placed in a growth chamber that had been adjusted to 37 C. After 24 hours, the radicle was starting to protrude from the seed coat. The seeds used in the experiment were at this stage of germination at the time of planting.

One corn seed was placed in an acetate tube cut 15.24 cm in length and each tube had an inside diameter of 2.54 cm. Soils were screened, homogenized, and allowed to air-dry for 24 h before beginning the bioassay. The tubes were capped on the lower end with a rubber cap with four holes cut in each cap. A small cotton ball was placed in the bottom of each tube to prevent soil from exiting the tube. The tubes were filled with approximately 80 g of soil, thus three replications could be achieved from one field sample. Once filled, they were placed in a tube rack and the tray was filled with deionized water until the water level was 1.0 cm above the cap, approximately 3.5 cm from the bottom of the tray. The tubes were allowed to soak until the deionized water was absorbed to the top of the soil. This procedure took approximately 1 h.

After thorough soaking, one germinated corn seed was placed in the center of the tube, with the radicle pointing downward. The seed was covered with soil and acid washed sand was used to fill the tube approximately 2 cm. The sand was moistened to prevent crusting. After covering with sand, black plastic was used to cover the entire bench area, approximately 15 cm above the tubes, to limit light interaction with the corn roots.

The corn plants were allowed to grow for 6 days and then harvested. The radicle length was measured and the data was subjected to analysis. A standard response curve was performed for each experiment. Technical grade chemicals were received with the following code names and purity: imazaquin (AC5105-10, 99,1% active) and imazethapyr (AC5561-85, 99.1% active), from American Cyanamid, and chlorimuron (DPX-F6025-108, 98.7% active), from DuPont.

The concentration range for imazaquin and imazethapyr was 0.0, 2.5, 10, 30, 50, and 100 ppb and was 0.00, 0.01, 0.02, 0.04, 0.05, and 0.10 ppm for chlorimuron. Soils treated with these concentrations were subjected to the same procedures as the field samples. The soil for the standard response curve was taken from the alleyways of each location. Each sample was replicated 15 times.

Results from the field samples were compared to that of the standard by fitting the root length to a model. The concentration for each treatment in the field was then determined and subjected to regression analysis. Three samples were entered into the bioassay from each field sample and the mean was determined. The values that were used for regression analysis were the mean of each field sample, yielding 4 data points. Regression of imazaquin and imazethapyr concentrations against time did not yield significant linear, curvilinear, or logarithmic models. Concentrations of imazaquin and imazethapyr were converted to the natural logarithm for a first-order model of herbicide dissipation which also accounted for more of the variation than any other model attempted. Actual concentration values were used for chlorimuron and these values were regressed over time. Analysis of covariance was also performed to make inferences about treatment means.

C. Results and Discussion

In 1989, soybeans at Knoxville and Springfield were not planted until late July which is later than the recommended planting dates. The University of Tennessee recommends that soybeans be planted no later than July 15th. Herbicide applications were made July 5, 1989 at Springfield and immediately after spraying, (2 cm) rainfall occurred. Severe erosion occurred and it is believed that much of the herbicides moved off the field with the runoff water and eroded soil. There were several gullies located within the field measuring 15-20 cm deep. Skips were noticed within the row and soybean establishment was estimated to be less than 50%. Soybeans were not planted until July 9th because of soil moisture conditions. Soil samples were not taken on day 0 or on day 8. Day 4 soil sampling was not taken the second year because research from the previous year's bioassay showed no significant difference in the amount of herbicide present between day 0 and day 4.

The soybeans at Knoxville in 1989 were planted on July 28, but unlike Springfield, an adequate stand was established; however, soybeans did not achieve normal heights. Rainfall (4 cm) was received within 7 days after application. Soil samples were taken as the previous year with the exception of day 4.

In 1989 at Milan, soybeans were planted on June 2 and heavy rainfall occurred immediately after planting. Phytotoxicity from imazaquin, imazethapyr, and chlorimuron was noted. Plant heights were taken 32 and 64 days after treatment (DAT) and were subjected to analysis. Expected phytotoxicity symptoms of these chemicals occurred on all treated plots regardless of soil pH (Table 1). Phytotoxicity appeared to be augmented by excessive moisture received after herbicide incorporation. This excessive moisture causes the herbicide molecules to be taken up more readily by the soybean plants (34). However, by 64 DAT, soybean plants in the imazaquin and imazethapyr plots had grown

Table 1. Herbicide effects on plant height of soybeans at Milan in 1989. Plant heights were measured 32 and 64 days after treatment[a].

	Plant Height	
Herbicide	32 DAT	64 DAT
	cm	
chlorimuron	27.464 c	48.251 c
imazaquin	27.464 c	80.250 b
imazethapyr	31.432 b	82.750 ab
control	35.969 a	86.250 a

[a] Means within a column followed by the same letter are not significantly different at the 5% level using Duncan's Multiple Range Test.

out of the stunting and the imazethapyr treated plots were not significantly different in height from the control plants. Chlorimuron- and imazaquin-treated plants were significantly shorter than control plants. Chlorimuron-treated plants were severely shortened and plants never recovered from the initial injury Soybean yields from the chlorimuron plots were extremely low (<1000 kg ha^{-1}).

Yields were affected both years by pH (Table 2). The plots with pH of 6.5 or higher out yielded low pH plots. Rainfall received from April to October at Springfield (35 cm) and Milan (30 cm) was extremely low and rainfall was not received within a two week period after treatment in 1988. It is evident that soil moisture plays an important role in yield and in allowing $Ca(OH)_2$ to go in solution (21).
Soil samples were taken following harvest in 1988 and apparently due to insufficient rainfall, most $Ca(OH)_2$ was not in solution. Therefore, less was needed in 1989 to adjust soil pH. At Knoxville in 1988, the entire test was irrigated within two days of $Ca(OH)_2$ application. In 1988, Knoxville received sufficient rainfall and being on a low site, soil moisture conditions remained adequate throughout the growing season. More $Ca(OH)_2$ was needed (approximately 5 T ha^{-1}) to adjust soil pH in the higher pH plots.

Yields at Milan in 1989 show very significant effects due to pH and soil moisture. Significant differences were observed among all four pH treatments. Yields were higher in the high pH treatments than in the low pH treatments.

The field at Knoxville was on an extremely low site. This proved to be advantageous in 1988 because of the lack of rainfall that

Table 2. Effects of Soil pH on Soybean Yield[a].

pH	Milan		Springfield	Knoxville
	1988	1989	1988	1988
	----------mean yields (kg ha^{-1})----------			
5.2	1843.91 a	1172.58 d	1602.46 a	3629.99 b
5.5	1470.14 a	1500.90 c	1497.10 a	3465.93 c
6.5	1524.40 a	1696.67 b	1529.98 a	3652.72 b
7.2	1201.86 a	1825.15 a	1712.19 a	3866.13 a

[a] Means within a column followed by the same letter are not significantly different at the 5% level using Duncans Multiple Range Test.

year. Yields at Knoxville were as high as 4500 kg ha^{-1} in some plots. Consequently, the location was undesirable in 1989 because of the excessive amount of rainfall which resulted in delayed soybean planting. Wet soil conditions delayed incorporation of hydrated lime needed to adjust pH in 1989. However, pH plots at Knoxville were extremely well defined in 1989. The soybean plants in the higher pH plots were readily distinguished from the lower pH plots. In the higher pH plots, the soybean plants were dark green, whereas the plants in the low pH plots were greenish-yellow. The difference between plots of 6.5 and 7.2 were distinguishable, but differences were not as great as with low pH plots.

Problems developed early at the Springfield location in 1988. Poor communication among the staff inadvertently resulted in incorporation of lime with the leveling bar lowered behind the Do-All. The lime was applied by hand to a specific area, but during incorporation, the lime appeared to be dragged away from the target area. With the lack of rain in 1988, it appeared soil pH was not affected significantly by spreading lime over the entire test, for pH measurements showed pH levels close to that desired. Soil samples were taken after harvest in 1988 and calculations were made to adjust the pH to the desired level as was done the previous year. Additional lime was applied in April of 1989. The pH measurements of 1988 appeared to be pure calcium measurements, for with the excessive amount of rainfall in 1989, the majority of the lime appeared to have been washed away. The addition of lime in 1989 did not raise soil pH to that desired, especially in the high (7.2) pH plots (yield and bioassay results for Springfield in 1989 are not shown).

In 1988 at Knoxville, limestone was applied in the form of $CaCO_3$. Screen size for this lime was 20, with 90% passing, and was applied with a Gandy fertilizer spreader. Rainfall was received in adequate amounts and soil samples were taken to measure the reaction change. These tests indicated that the majority of the limestone had not gone into solution. A decision was made to apply hydrated lime in an attempt to decrease the reaction time. Hydrated lime usually reacts within two weeks if significant rainfall occurs. After the hydrated lime had been applied, it was incorporated in two directions while not allowing the lime to move outside the plots. Results from soil samples indicated the lime reacted yielding a soil pH close to that desired.

Greenhouse Bioassay

Imazaquin and Imazethapyr. The greenhouse portion of this research was performed in 1989 at the University of Tennessee, Knoxville, in the greenhouse on the Agricultural Campus. The greenhouse conditions throughout the year were quite variable. Average daily temperatures in the middle of the summer were in excess of 37 C. This apparently had no effect upon the outcome of the bioassay.

The analysis of soil residues from Knoxville in 1988 showed better results than any other location. The moisture levels at Knoxville were adequate throughout the growing season. Imazaquin residues were found until 128 DAT. More imazaquin residue was found in the low soil pH

(5.2) than with the high pH (7.2). A discernible reduction in concentration occurred by 16 DAT. However, in the low pH soil, imazaquin concentration was still quite high even at day 64. Apparently as the pH increases from 5.2 to 6.5, imazaquin dissipates at an increasing rate. At day 128, imazaquin was more prevalent in the low pH soil (5.2).

Results from Knoxville during 1988 show there was considerable variation that cannot be explained. Low R^2 values indicate that most of the variation in concentration was not accounted for in the model. From the regression lines (Figure 1), there appears to be a trend toward a slower dissipation of imazaquin at low soil pH's of 5.2 and 5.5. As the pH increases to 6.5 and 7.2, the concentration decreased more rapidly.

Irrigation (3.8 cm) was supplied 4 days after herbicides were sprayed in 1988. Rainfall occurred 9 days after soybeans were planted, so adequate soil moisture was available for herbicide activation. Initially, herbicide recovery at day 0 was approximately 75%. Imazaquin persistence appeared to remain steady throughout the growing season, with the exception of the high pH plots. This could be due to the amount of soil moisture keeping imazaquin in solution. Being on a low site, the soil moisture was adequate throughout the growing season, even though only 3 cm was received in June of 1988.

Imazethapyr degradation rates remained fairly constant with the exception of pH 7.2 (Figure 2), but concentrations decreased with time. There appear to be no significant differences at pH 5.2, 5.5, or 6.5,

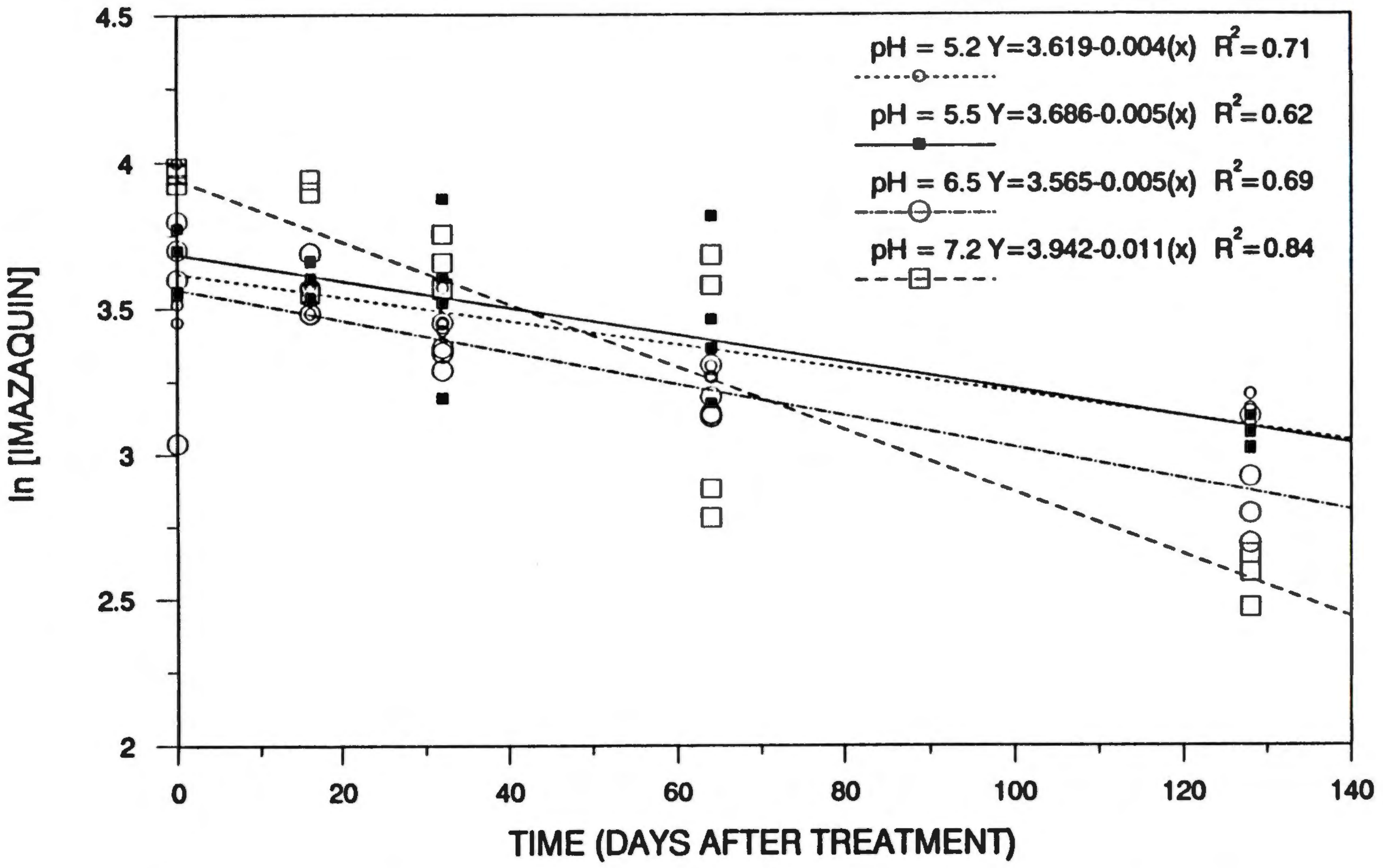

Figure 1. Linear regression showing the effect of soil pH on the natural log of the imazaquin concentrations at Knoxville during 1988 sampled over varying time intervals following application. Plotted values are the means of three replicates.

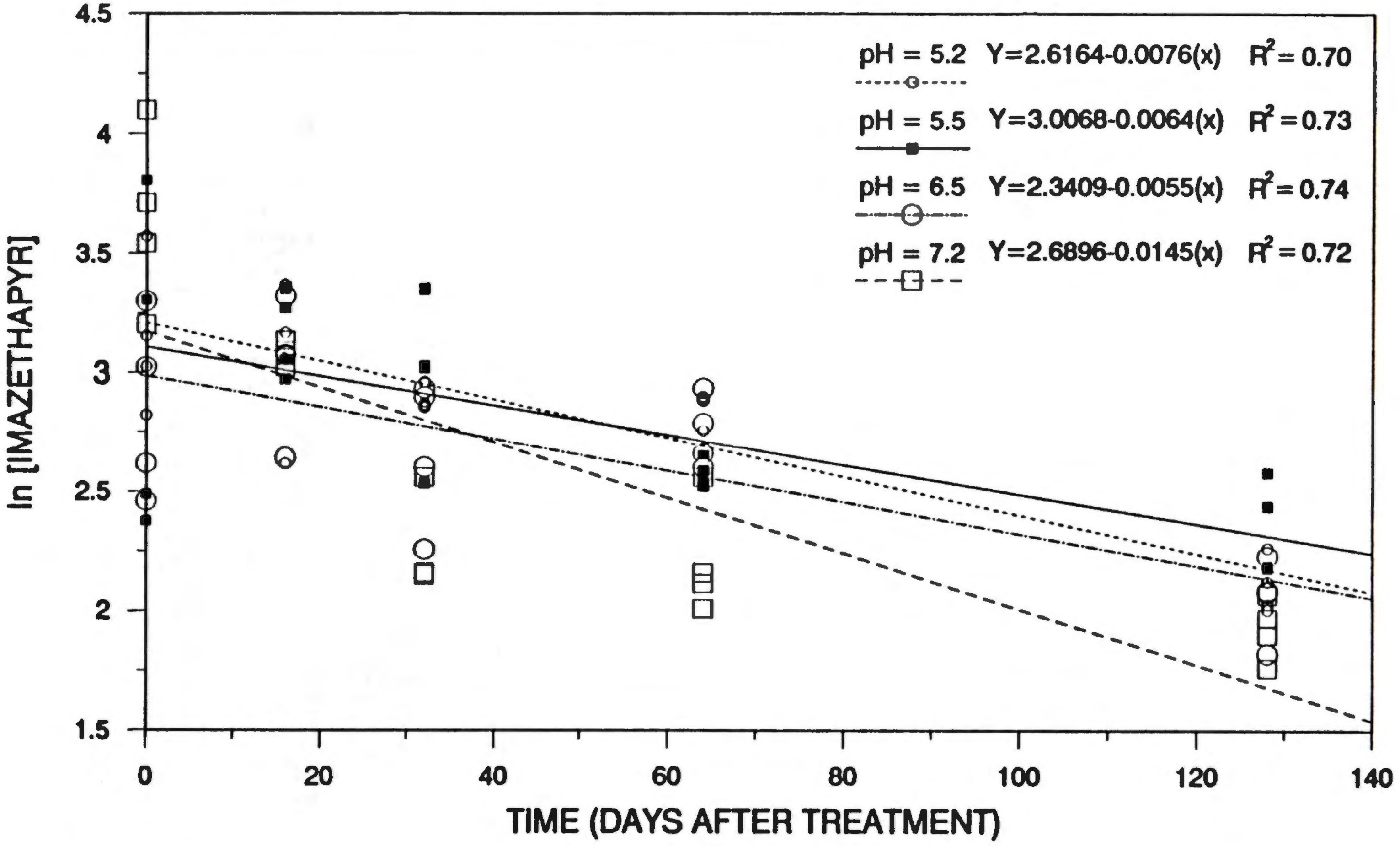

Figure 2. Linear regression showing the effect of soil pH on the natural log of the imazethapyr concentrations at Knoxville during 1988 sampled over varying time intervals following application. Plotted values are the means of three replicates.

but at pH 7.2, persistence decreases. Variation was quite high in this analysis as revealed by the low R^2 values.

At Milan in 1988, rainfall was not received during the month of June. Imazaquin recovery was approximately 90% at day 0 of sampling. Low pH plots retained more imazaquin than did the high pH plots. Lower concentrations at 128 DAT were detected in the high pH plots (Figure 3). Low R^2 values again indicate the large amount of variation not accounted for by the model.

Imazethapyr recovery was approximately 85% at 0 DAT. Apparently there was a trend for greater imazethapyr persistence at low soil pH, but significant differences among pH's was not observed during analysis (Figure 4).

At Springfield in 1988, a drought situation occurred and herbicides did not perform appropriately. Significant rainfall did not occur until twenty days after application. Imazaquin was the only herbicide evaluated that was found in significant amounts in the soil bioassay. Apparently soil moisture was a significant factor in the outcome of the imazaquin bioassay. The results (Figure 5) show that pH had no significant effect on imazaquin concentration. The results of all four pH's were very close, which is unlike the results from other locations. Low soil moisture is believed to be the cause of the dissipation rates to be grouped so closely. Imazethapyr results were near the same as with imazaquin (Figure 6). All four pH's were grouped closely indicating no difference in herbicide persistence due to soil pH. However, low soil moisture and poor lime incorporation are factors

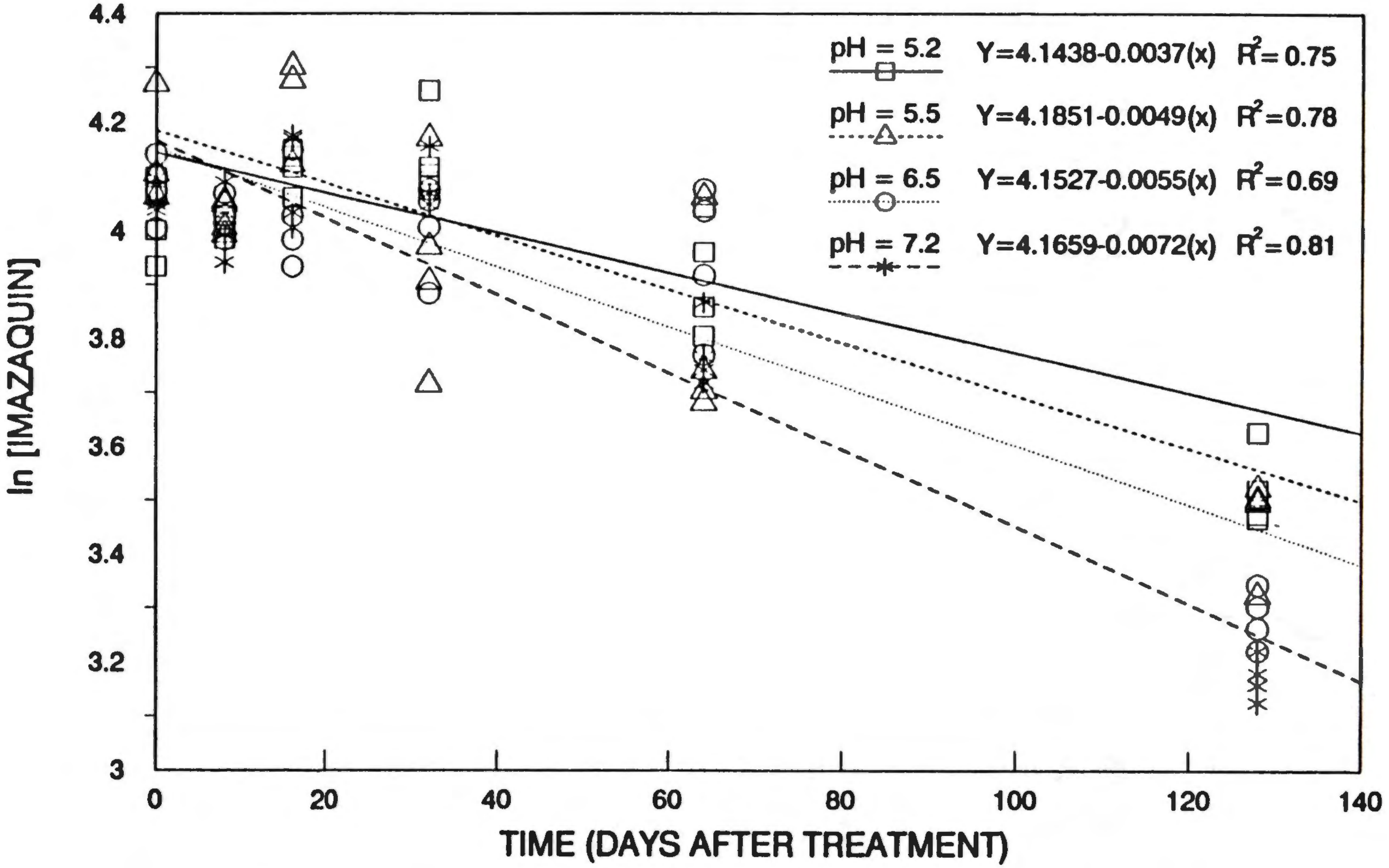

Figure 3. Linear regression showing the effect of soil pH on the natural log of the imazaquin concentrations at Milan during 1988 sampled over varying time intervals following application. Plotted values are the means of three replicates.

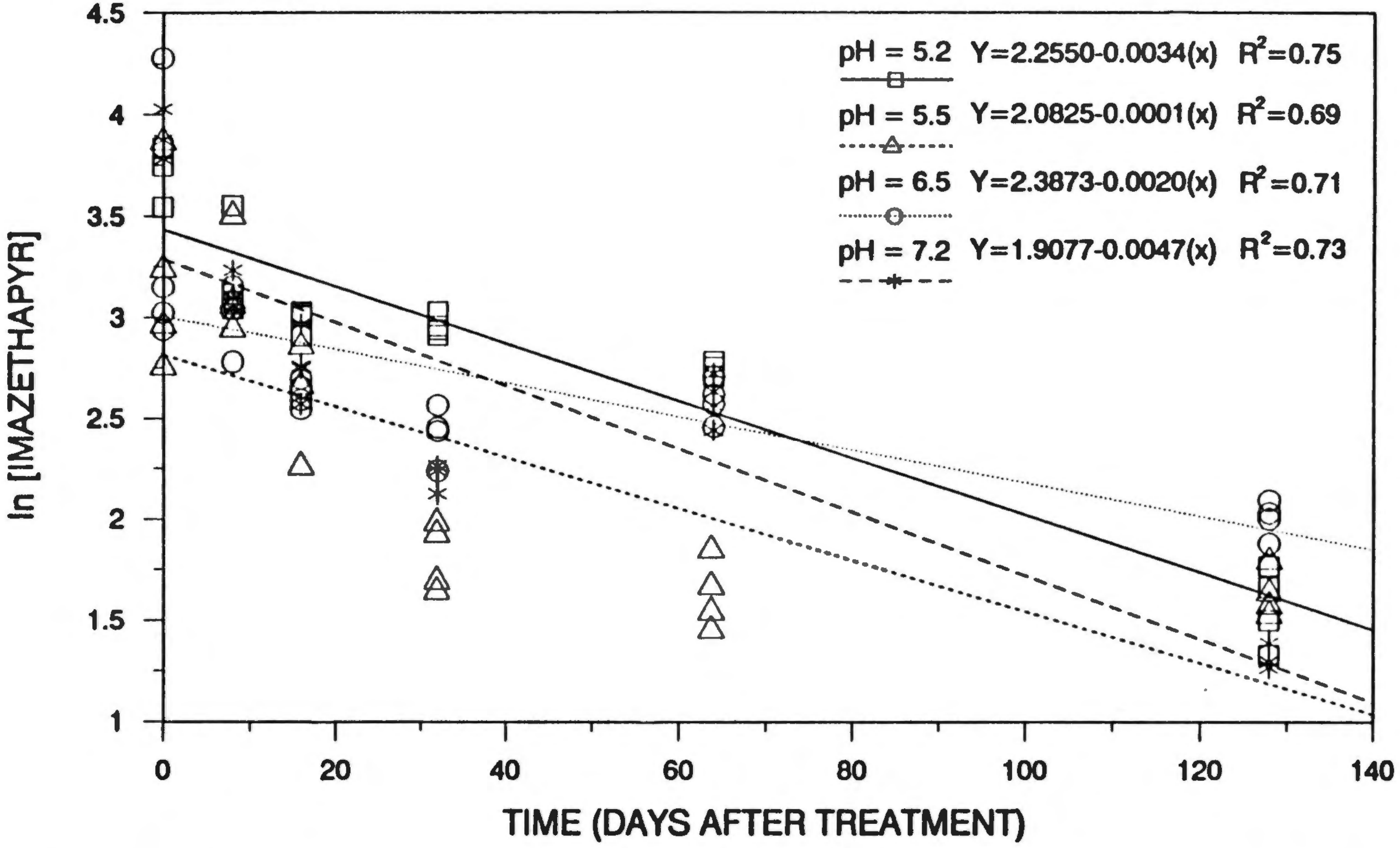

Figure 4. Linear regression showing the effect of soil pH on the natural log of the imazethapyr concentrations at Milan during 1988 sampled over varying time intervals following application. Plotted values are the means of three replicates.

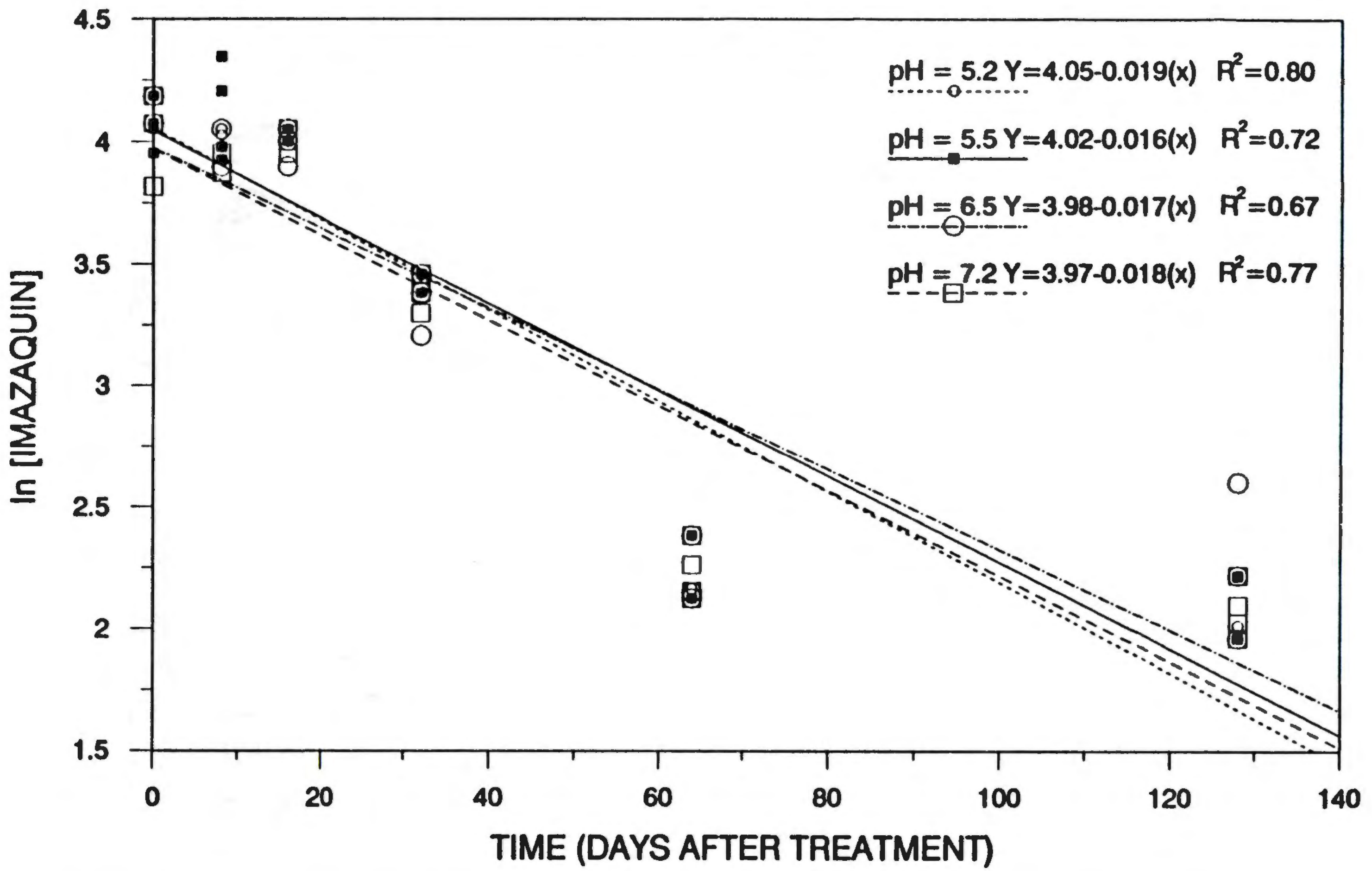

Figure 5. Linear regression showing the effect of soil pH on the natural log of the imazaquin concentrations at Springfield during 1988 sampled over varying time intervals following application. Plotted values are the means of three replicates.

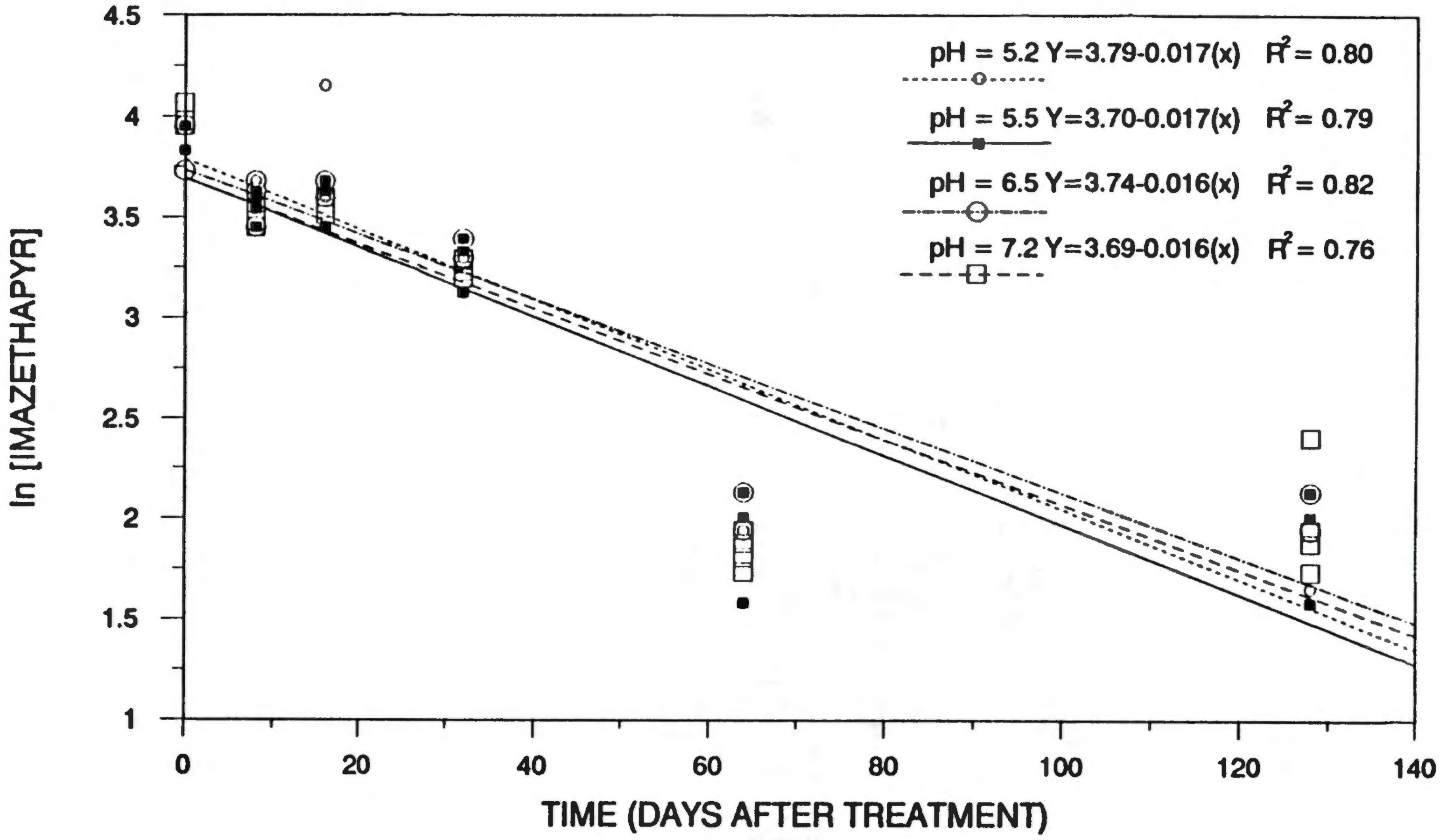

Figure 6. Linear regression showing the effect of soil pH on the natural log of the imazethapyr concentrations at Springfield during 1988 sampled over varying time intervals following application. Plotted values are the means of three replicates.

to be considered. Initial herbicide recovery was 90% for both imazaquin and imazethapyr.

The environmental conditions in 1989 were completely opposite of those in 1988. Wet soil conditions prevented early application of lime to adjust pH's, as well as soybean planting, with the exception of Milan. Rainfall in 1989 set several records for Springfield and Milan. Field studies were affected by the large amounts of rainfall.

The Knoxville location provided good results in 1989 even though soybeans were planted on July 28. Regression analysis (Figure 7) indicates low R^2 values. Imazaquin was more persistent in the low pH plots of 5.2, 5.5, and even 6.5, with more dissipation in the high pH plot. Herbicide recovery was near 100% at day 0. The trend for imazaquin persistence in low soil pH's is evident from the bioassay.

Imazethapyr concentrations were not as strongly affected by soil pH as was imazaquin (Figure 8). However, there appears to be a higher concentration of imazethapyr with the lower soil pH's. Both imazaquin and imazethapyr appear to dissipate less in soils with low pH values. Both 1988 and 1989 results from Knoxville show more persistence of the imidazolinones at low soil pH.

The same results were observed at Milan in 1989. The trend for imazaquin to be more persistent at low pH and less at higher pH was evident (Figure 9). Again, imazethapyr did not seem to be affected by soil pH (Figure 10).

At Springfield in 1989, torrential rains occurred immediately after herbicide application that prevented incorporation. The rainfall, it was hoped, would incorporate the herbicide, similar to a

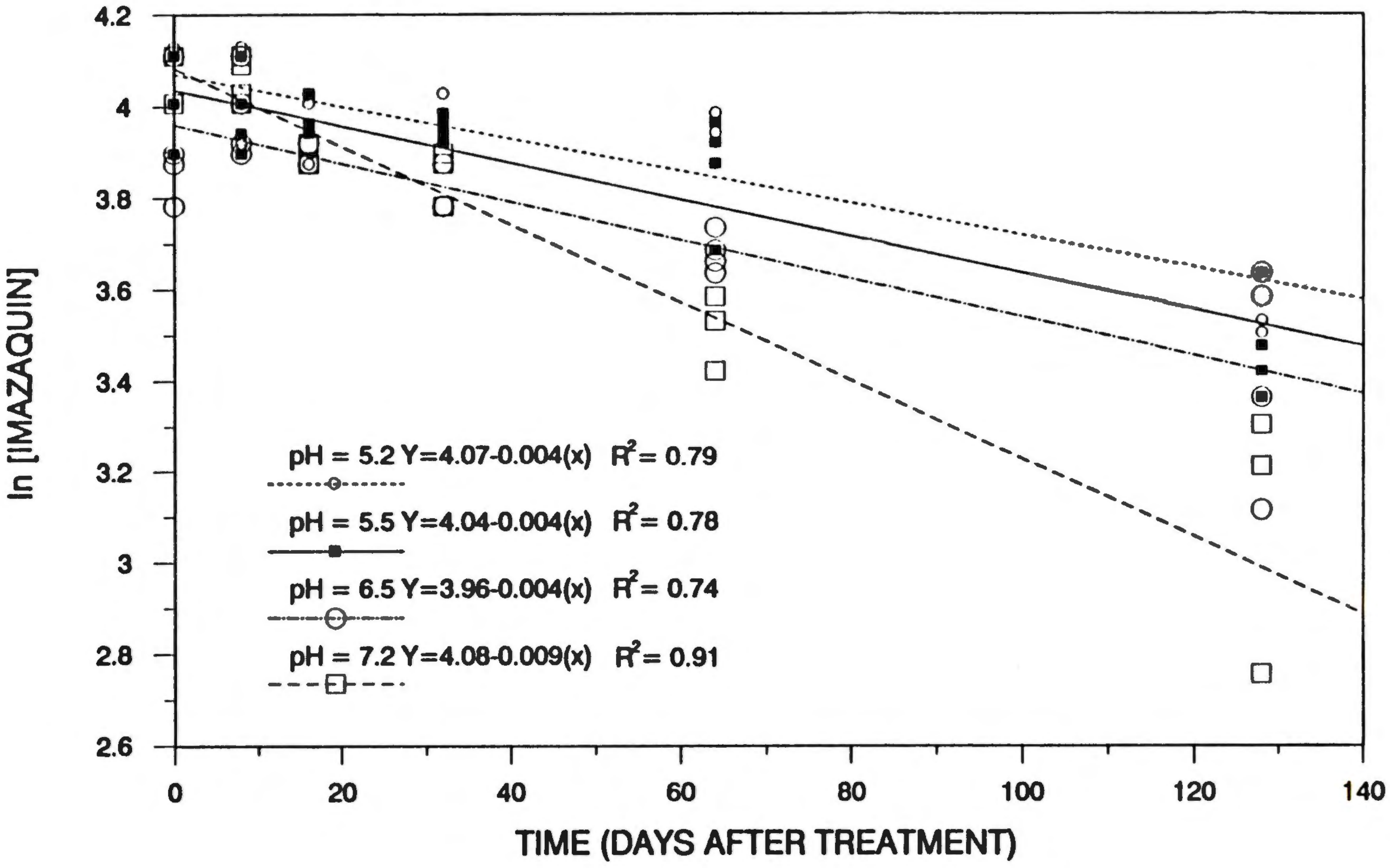

Figure 7. Linear regression showing the effect of soil pH on the natural log of the imazaquin concentrations at Knoxville during 1989 sampled over varying time intervals following application. Plotted values are the means of three replicates.

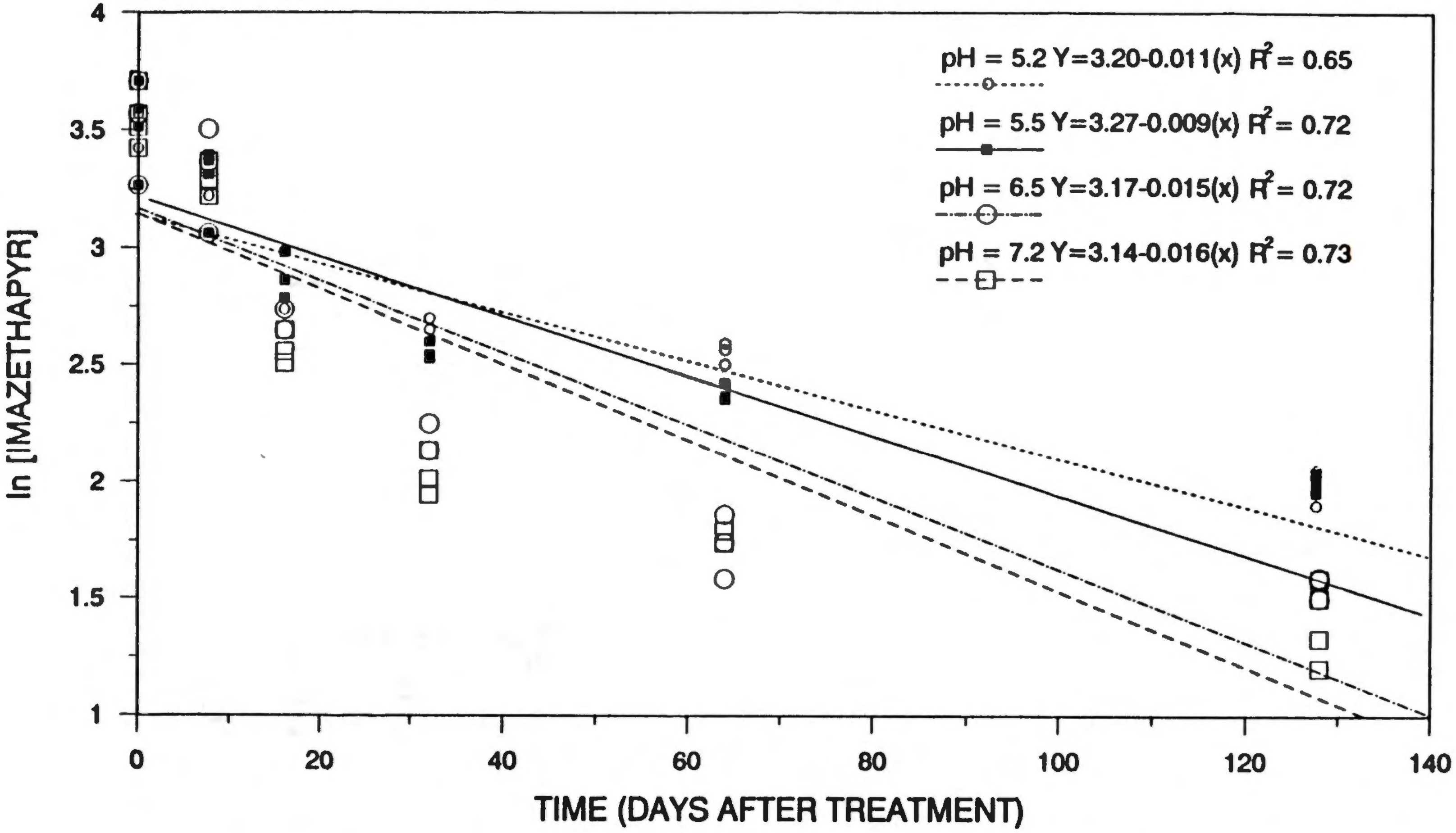

Figure 8. Linear regression showing the effect of soil pH on the natural log of the imazethapyr concentrations at Knoxville during 1989 sampled over varying time intervals following application. Plotted values are the means of three replicates.

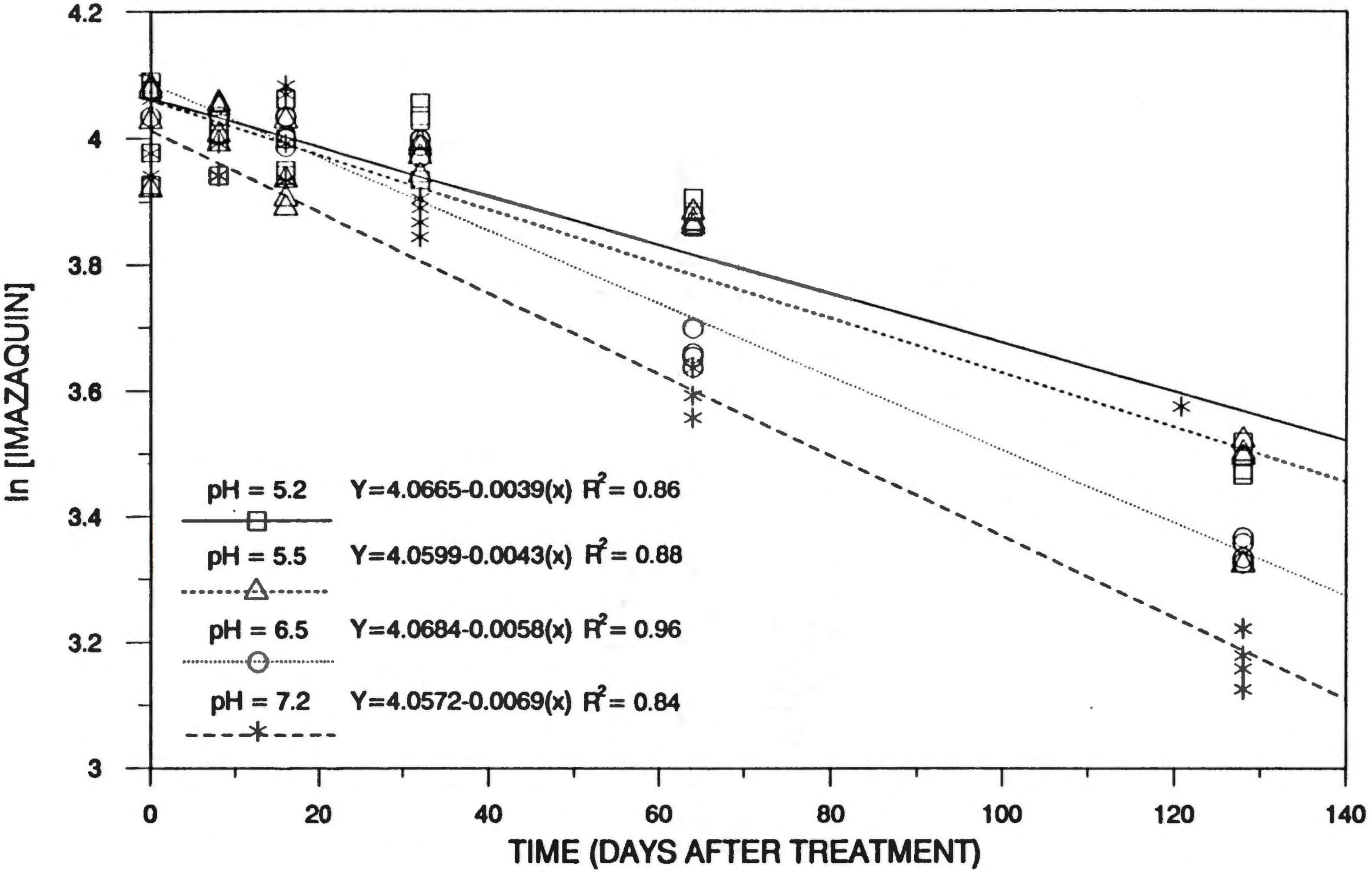

Figure 9. Linear regression showing the effect of soil pH on the natural log of the imazaquin concentrations at Milan during 1989 sampled over varying time intervals following application. Plotted values are the means of three replicates.

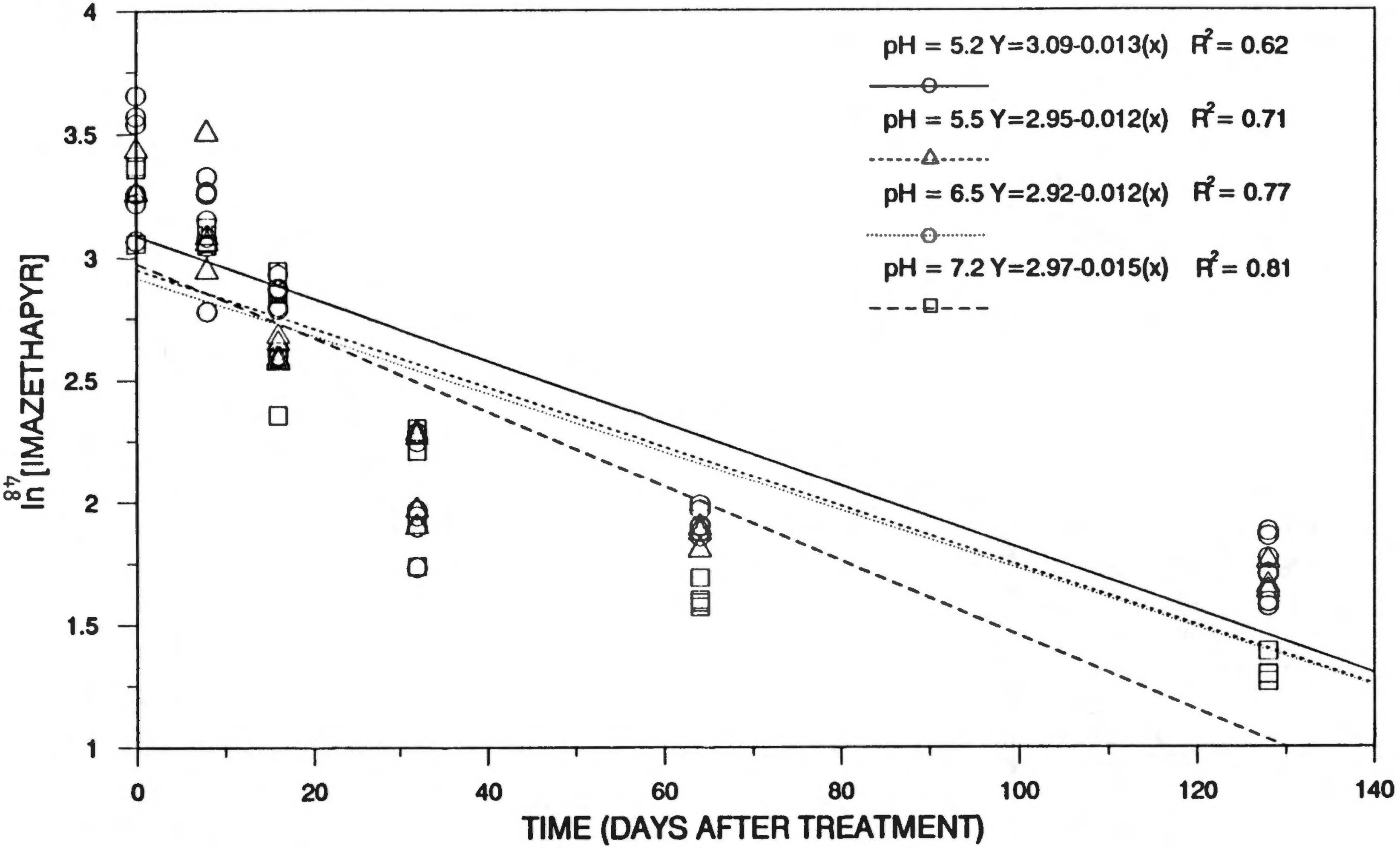

Figure 10. Linear regression showing the effect of soil pH on the natural log of the imazethapyr concentrations at Milan during 1989 sampled over varying time intervals following application. Plotted values are the means of three replicates.

herbigation application. However, bioassays of that year proved just the opposite. In most of the other bioassays performed, imazaquin at each pH was at least recognizable 16 DAT. In 1989, samples at 16 DAT showed no significant amount of imazaquin, imazethapyr, or chlorimuron present. Heavy rains and severe erosion are believed to be the elements responsible for moving the herbicide from the site of application. Data from Springfield in 1989 are not shown.

Chlorimuron. Chlorimuron was most persistent in soils with a high pH value. Chlorimuron is a weakly acidic compound and in soils with pH higher than the pK_a value, chlorimuron would be expected to be more persistent.

Useable results were obtained at Knoxville in 1988 and 1989 and at Milan in 1989. Drought conditions occurred at Milan and Springfield during 1988 and are believed to be the reason for little chlorimuron persistence. Herbicides having more than one route of dissipation can be expected to have fewer carryover problems (26). With the lack of rainfall, the added lime never reached equilibrium at these locations even though soil pH analysis measured the high pH to be 7.2. However, the Knoxville location received irrigation (3.8 cm) and adequate rainfall in 1988 and the location was on a low site, thus soil conditions were moist throughout the growing season.

At Knoxville in 1988, chlorimuron persistented in the plots with a pH value of 7.2 (Figure 11). As the soil pH decreased to 6.5, chlorimuron degradation increased and the increase was about the same

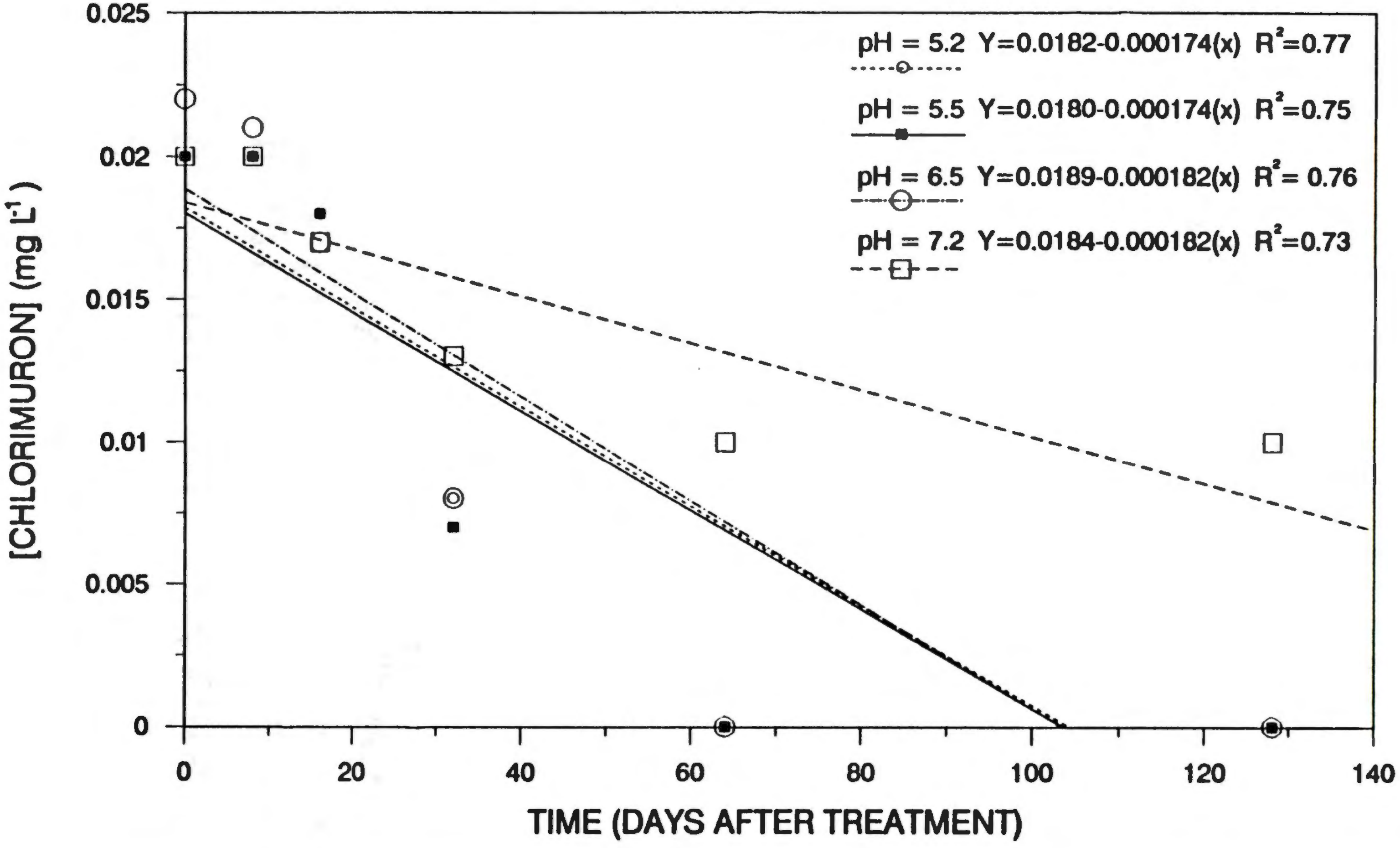

Figure 11. Linear regression showing the effect of soil pH on chlorimuron persistence at Knoxville during 1988 sampled over varying time intervals following application. Plotted values are the means of three replicates.

with the lower soil pH's. In the pH plots of 5.2, 5.5, and 6.5, no chlorimuron was detected in the bioassay 32 DAT. Chlorimuron is believed to have dissipated to an undetectable level by this date. Low R^2 values were again seen and variation seems to be due to several factors that were unaccounted for in the model.

At Milan and Springfield in 1988, chlorimuron was not detected at day 32. Good recovery (95%) of chlorimuron was, however, noted at day 0 (Figure 12). The situation at Milan is believed to be the outcome of a very dry year. Added lime is assumed not to have gone into soil solution. Chlorimuron, being a weakly acidic herbicide, is believed to have dissipated by this time, since soil pH was 7.2. Earlier studies have shown the half life of chlorimuron to be divided into two phases, the early and late phase (62). The early phase was reported to be around 20 to 30 days. This is consistent with the data presented here, since chlorimuron was not detected in the bioassay 32 DAT. Results from Springfield in 1988 were almost the same as Milan in 1988. The dry soil moisture conditions as well as the incorporation of lime apparently had a significant effect upon chlorimuron persistence. At Milan, 90% of the chlorimuron was recovered at day 0, but at 32 DAT, no detectable level of chlorimuron was observed (Figure 13). There appears to be an abrupt change in the amount of chlorimuron between 16 and 32 DAT. This is believed to be the first phase of chlorimuron degradation.

Even though spring rains prevented early soybean planting at Knoxville in 1989, chlorimuron persistence was observed. Apparently higher soil moisture conditions differentiate between 1988 and 1989.

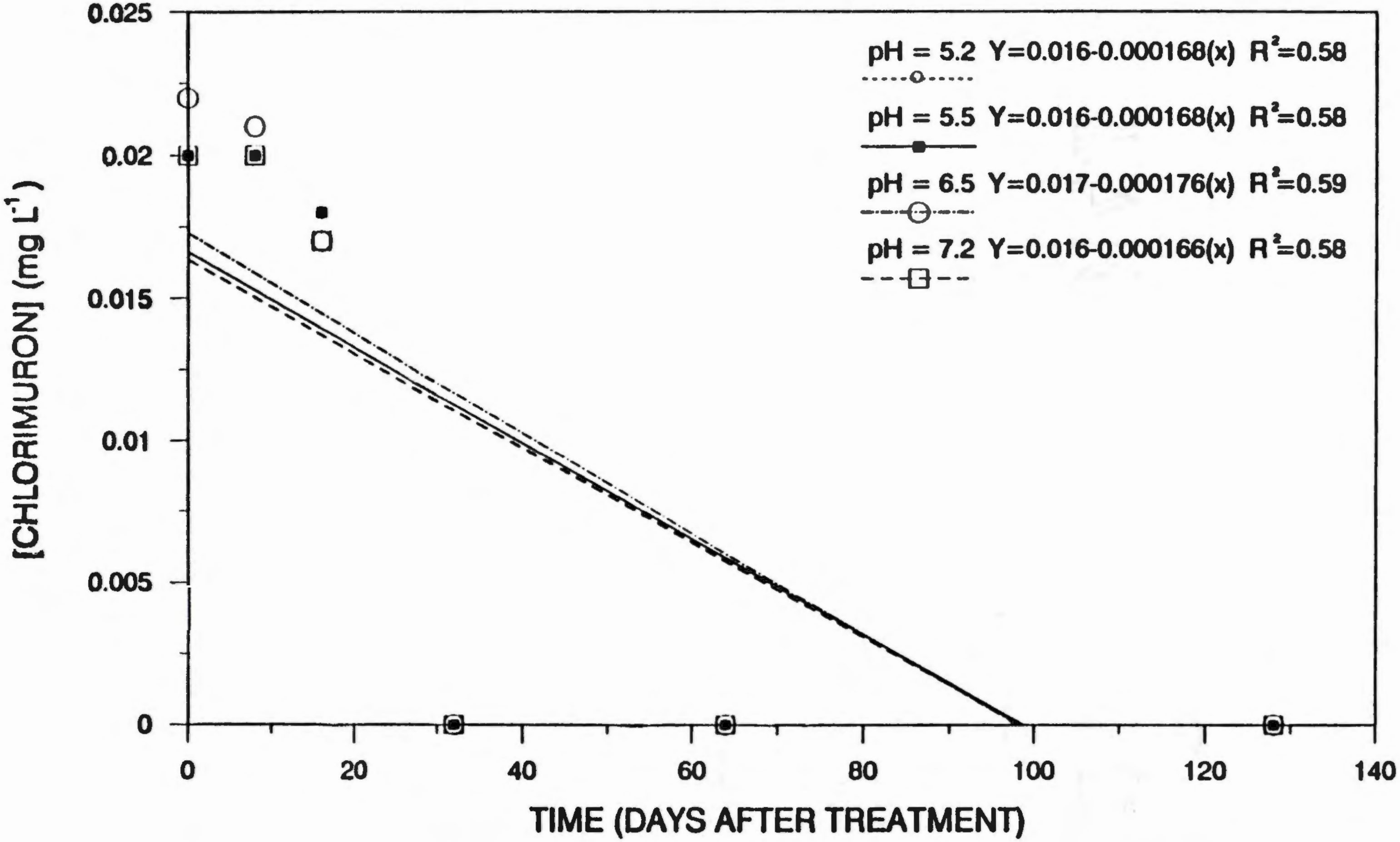

Figure 12. Linear regression showing the effect of soil pH on chlorimuron persistence at Milan during 1988 sampled over varying time intervals following application. Plotted values are the means of three replicates. Regression lines for pH treatments 5.5 and 7.2 are superimposed.

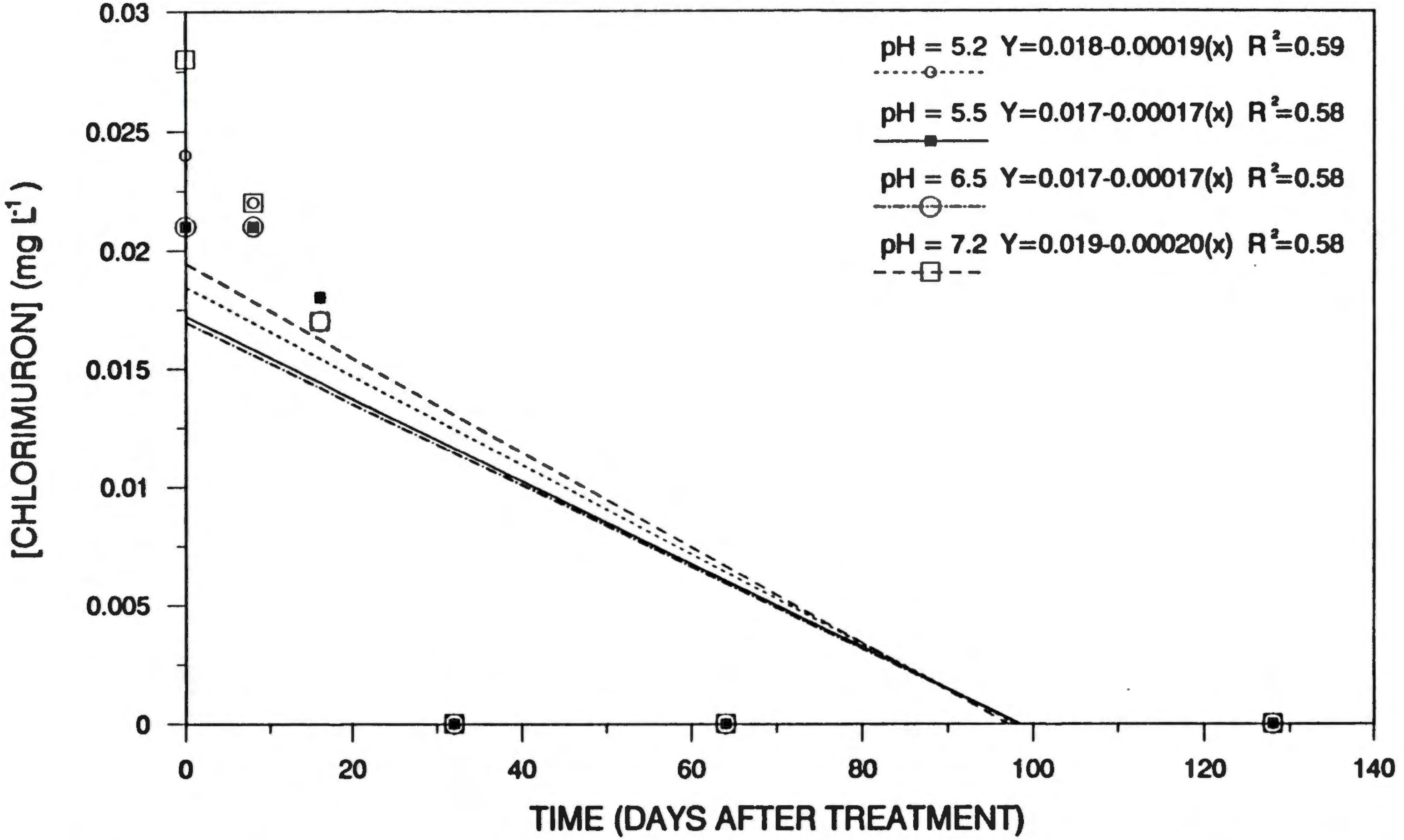

Figure 13. Linear regression showing the effect of soil pH on chlorimuron persistence at Springfield during 1988 sampled over varying time intervals following application. Plotted values are the means of three replicates.

Soil pH was altered in April and soil pH measurements in June revealed the majority of the lime had reacted and was in equilibrium. The excessive amount of rainfall postponed planting of soybeans until the last week of July. The soil remained adequately moist throughout the growing season. It would seem soil conditions were conducive to degradation throughout the sampling season in 1989. Chlorimuron concentrations were detectable at pH 5.2 and 5.5, 32 DAT (Figure 14). However, by 64 DAT, no detectable level of chlorimuron was found. At pH 7.2, chlorimuron was detected at 128 DAT. Higher concentrations of about 0.01 ppm were found at day 128. There appears to be a significant difference between chlorimuron persistence at pH 7.2 and at 5.2, 5.5, and 6.5. Again variation was high as is revealed by the low R^2 values.

The same situation occurred at Milan in 1989. Rainfall was received during the growing season and chlorimuron persisted in the high pH plots (Figure 15). Chlorimuron was detected at pH 6.5 at day 64 but was not detected at day 128. About 0.01 ppm was detected in the high pH plots at day 128. Higher R^2 values were obtained that cannot be explained. Early planting, early application of chlorimuron, and adequate soil moisture throughout the growing season are some factors that may be responsible for the better fit of the regression lines.

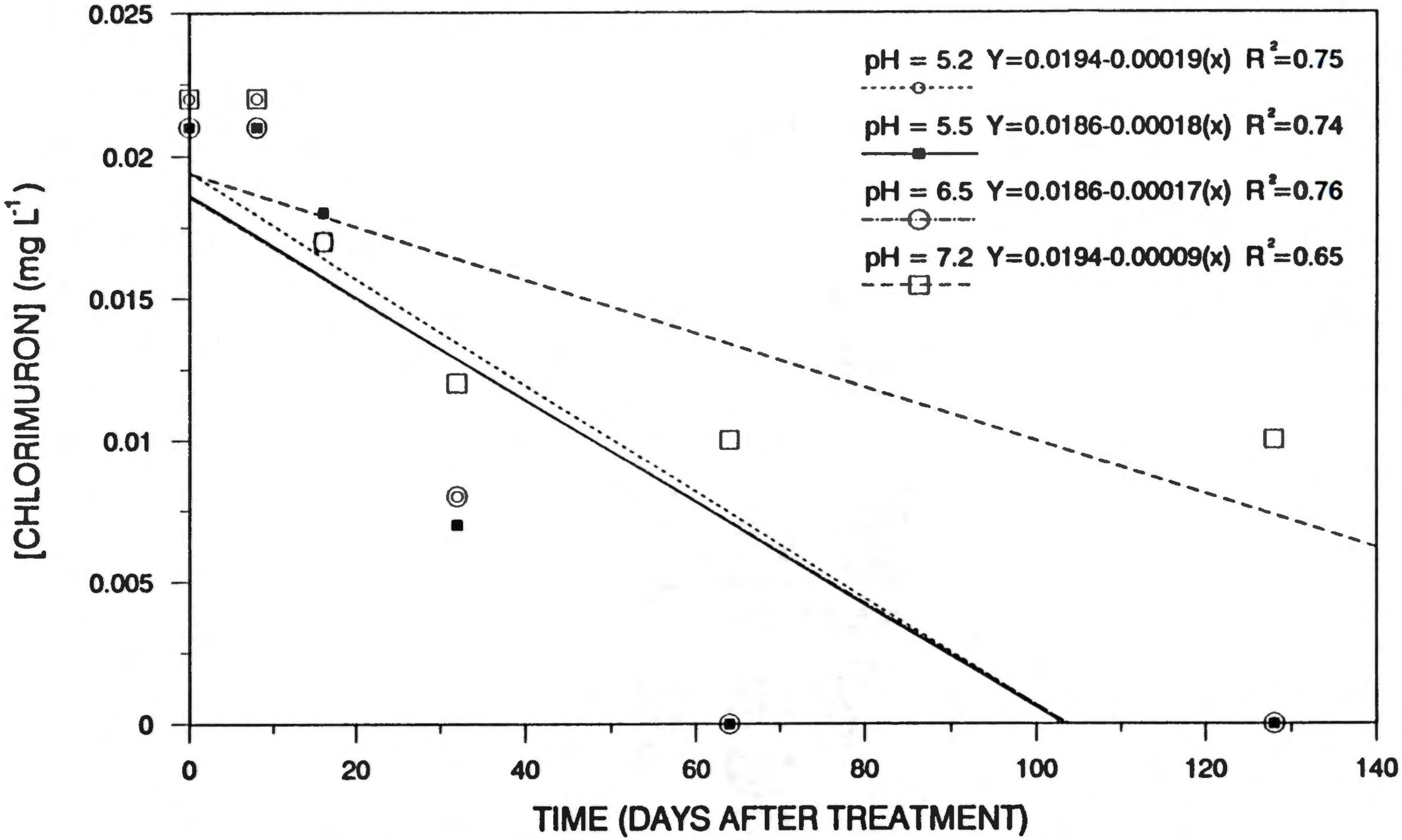

Figure 14. Linear regression showing the effect of soil pH on chlorimuron persistence at Knoxville during 1989 sampled over varying time intervals following application. Plotted values are the means of three replicates.

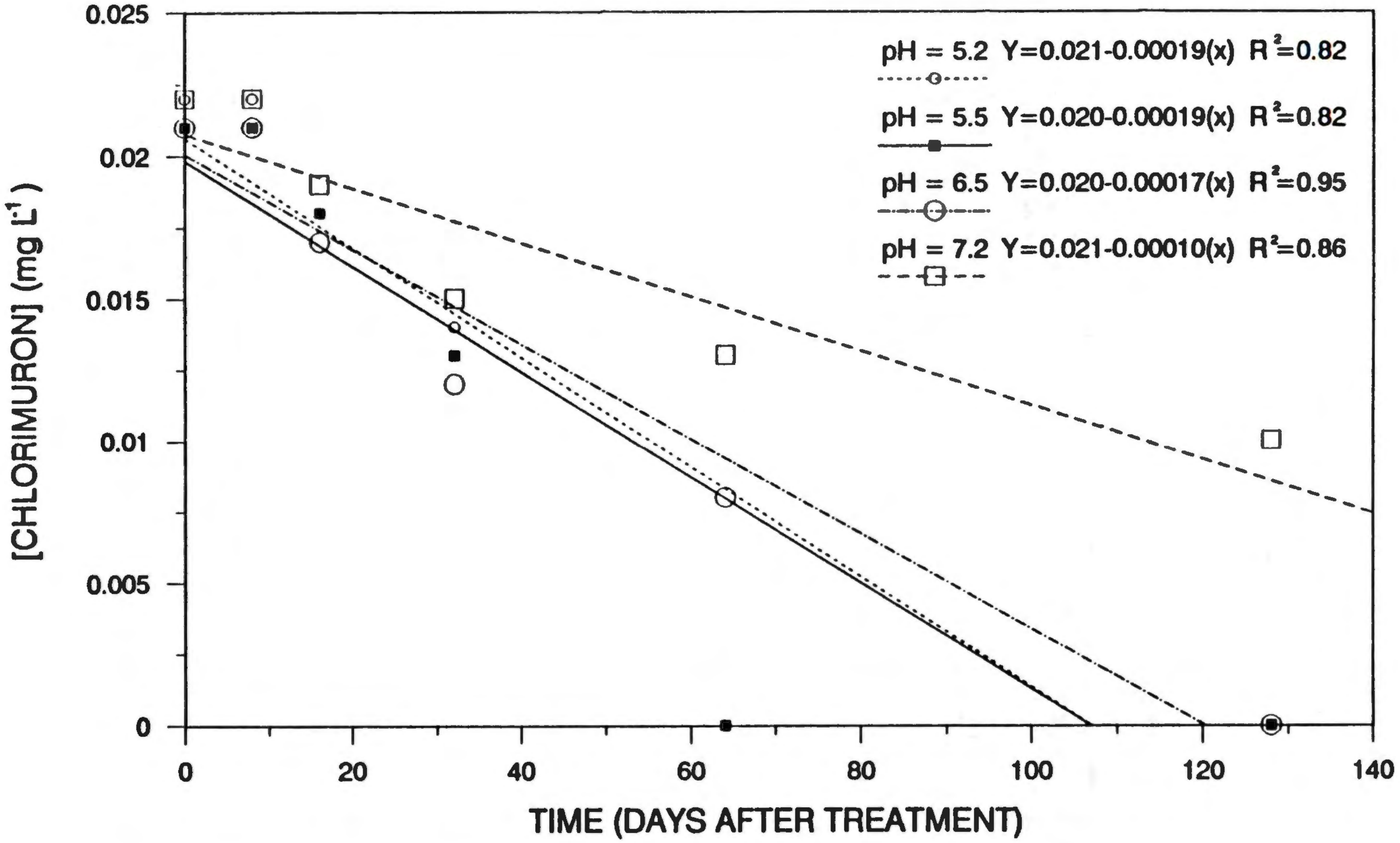

Figure 15. Linear regression showing the effect of soil pH on chlorimuron persistence at Milan during 1989 sampled over varying time intervals following application. Plotted values are the means of three replicates.

CHAPTER IV

GENERAL SUMMARY

The primary objective of this research was to evaluate the effects of varying soil pH's on the persistence and carryover of imazaquin and imazethapyr, both imidazolinones, and chlorimuron, a sulfonylurea, all applied pre-plant incorporated. All of these herbicides are available for use by the farmer.

Low R^2 values were obtained indicating variation in this experiment that was not explained by the model. There was considerable variation that occurred within each experimental unit. Adverse weather conditions, poor incorporation, and perhaps the bioassay itself are possible explanations for some of the variation. The greenhouse conditions for the bioassay could be an important factor causing high variation. Since there was significant variation, perhaps more replications should be performed to obtain a better idea of herbicide persistence.

There appears to be an overall trend at all locations both years with these herbicides. Herbicide dissipation increased from 0 to 32 DAT for both imazaquin and imazethapyr at all locations according to the regression lines. After day 32, dissipation was steady for imazaquin at pH 5.2 and 5.5. Imazethapyr appears in this study to decrease at an increasing rate (0 to 32 DAT), especially in 1989. This may have been due to the excessive amount of soil moisture. Imazethapyr has a high leaching capacity in soils and is much more soluble in water than imazaquin (1400 vs. 60 ppm). Since the soil was sampled to a depth of 15 cm, the data presented are not sufficient to make conclusions on herbicide movement in the soil, but it appears soil moisture plays an important role in the persistence of these herbicides. Corn roots were more tolerant of imazethapyr than imazaquin. At 0 DAT, both herbicides caused significant radicle length reduction, but by 8 and 16 DAT, root extension was inhibited more by imazaquin than by imazethapyr. Comparable concentrations for the same expression of symptoms was 35 ppb and 27 ppb for imazaquin and imazethapyr, respectively. Imazethapyr shows less potential for carryover into the next season at any soil pH and the reason appears to be related to the water solubility of imazethapyr.

The chlorimuron study showed that as the pH was increased from a low (5.2) to a high pH (7.2), the amount of chlorimuron present decreases at a decreasing rate. There appears therefore, to be a greater chance of chlorimuron persistence and possible carryover potential into the next crop in soils with high pH. Chlorimuron was undetected in low soil pH's at 32 DAT and in some cases was not detected in the high pH's. This appears to be due to the amount of soil moisture that is received early in the growing season. Studies from Milan and Springfield in 1988 show no detectable level of chlorimuron at any soil pH after day 32. These locations received very little rainfall that year.

Regressions of imazaquin concentration against time after the initial treatment showed that imazaquin was more persistent in soils of pH 5.2 and 5.5 than the other pH's studied. Collectively, the results of this series of experiments illustrate that soil pH does influence the carryover potential of these PPI herbicides. Significant amounts of variation can only lead to a speculation that there is a trend for the imidazolinone herbicide imazaquin to be persistent at low soil pH's, and sulfonylureas, like chlorimuron, to be more persistent at higher pH's. Imazaquin and imazethapyr are weakly acidic herbicides that are amphoteric compounds because they contain both acidic and basic functional groups. Protonation of the imidazolinone nitrogen may occur at low pH levels thus rendering a positively charged molecule. This would probably lead to cationic bonding to soil at low pH levels. Chlorimuron is also weakly acidic and at pH values higher than the pK_a value (4.2), the molecule is primarily anionic, and a potential for carryover into the next crop may occur. This is in agreement with Schmitz et al. (61) who concluded that chlorimuron is more persistent at high pH's. All of these factors including environmental conditions surely have an affect on the persistence of all three herbicides. It would be safe to assume that if both classes of these herbicides are to be used for weed control in soybeans, the soil pH should be near the optimum level. If this optimum pH level is not achieved, then there is a definite potential for these herbicides to carryover and cause significant damage to rotational crops.

The data reported here is in agreement with other investigators showing that the imidazolinones, especially imazaquin, are more persistent at low pH, and the sulfonylyureas are more persistent at high pH (17, 35). Further investigation is needed to determine all causes of carryover potential. Perhaps studies involving soil pH, soil moisture, application methods, and differing soil properties should be investigated collectively. These herbicides have great potential in weed control when used according to label specifications. This research, along with other research, indicates they will have a definite impact on weed science.

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APPENDIXES

APPENDIX A

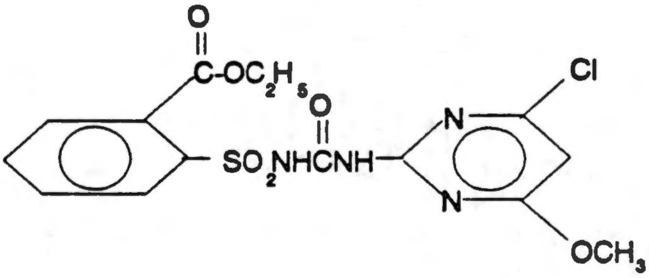
COMMON AND CHEMICAL NAMES OF HERBICIDES

APPENDIX A

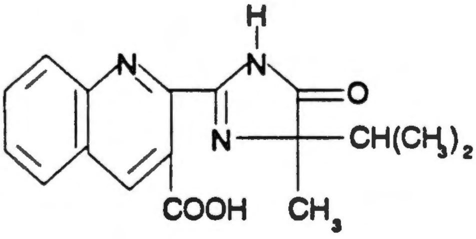
Table A-1. Common and chemicals names of herbicides used in this study

COMMON/TRADE NAME	CHEMICAL NAME
acifluorfen (Blazer)	sodium 5-[2-chloro-4-(trifluoromethyl)-phenoxyl]-2-nitrobenzoate
bentazon (Basagran)	3-isopropyl-1*H*-2,1,3-benzothiadiazin-(4)-3*H*-one-2,2-dioxide
chlorimuron (Classic) (DPX F-6025)	2-[[[[(4-chloro-6-methoxy-2-pyrimidinyl)amino]carbonyl] amino]sulfonyl]benzoic acid
imazaquin (Scepter) (AC-252,214)	2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1*H*-imidazol-2yl]-3-quinolinecarboxylic acid
imazethapyr (Pursuit) (AC-263,499)	± 2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1*H*-imidazol-2yl]-5-ethyl-3-pyridinecarboxylic acid
sethoxydim (Poast)	2-[1-(ethoxyimino)butyl]-5-[2-(ethylthio)propyl]-3-hydroxy-2-cyclohexen-1-one

Chlorimuron



Imazaquin



Imazethapyr

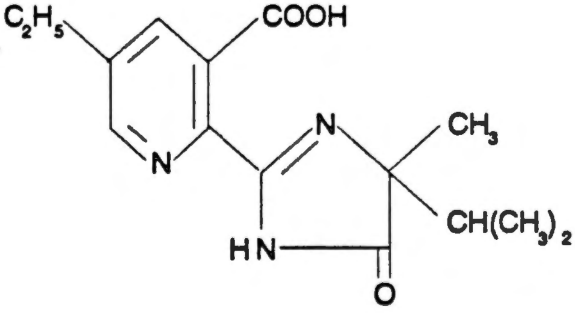


Figure 16. Structural formulas of selected herbicides.

APPENDIX B

RAINFALL DATA

APPENDIX B

Table B-1. Rainfall data at Knoxville in 1988.

DATE	APRIL	MAY	JUNE	JULY	AUG	SEPT
	(cm)					
1	0.10					
2	0.74					
3	0.08			0.05		
4	0.33	0.89		0.64		0.31
5		3.25			2.74	
6	0.33					
7	0.13				0.38	
8	0.25					
9		0.45				
10		0.41	1.60			
11		0.94				0.03
12	0.13			1.96	0.31	
13	0.13			0.05		0.18
14				5.21		
15						
16	0.79	0.03				
17		1.04				2.01
18	0.99			0.28		0.51
19	2.03					
20					1.14	
21			0.03	0.13	0.10	
22				3.43		
23		0.10		0.20		
24	0.28	0.46		0.18	0.25	
25		1.65	0.56			0.87
26			0.61			
27				0.10		
28				0.36		
29						
30			0.23			
32						
TOTAL	6.31	9.23	3.04	12.63	4.92	3.91

Table B-2. Rainfall data at Milan in 1988.

DATE	APRIL	MAY	JUNE	JULY	AUG	SEPT
	---------------------------(cm)-------------------------					
1						
2				2.69	0.08	
3						2.41
4						
5	0.25					
6						
7						
8		3.50				
9						
10				0.28		
11						0.05
12						
13				8.97		
14						
15						
16						
17	1.75					
18						
19						
20						
21					2.03	
22		3.96				
23						
24		1.25		7.24		
25						
26						
27						
28						
29						
30				5.46		
31						
TOTAL	2.00	8.71	0.00	24.64	2.11	2.46

Table B-3. Rainfall data at Springfield in 1988.

DATE	APRIL	MAY	JUNE	JULY	AUG	SEPT
			(cm)			
1	3.15					
2	1.42					
3	0.97				0.38	
4	0.33	1.70				4.01
5		0.33				
6	1.09					
7					0.51	
8						
9		0.99	0.23			
10		0.43		1.96	0.13	
11					0.25	0.05
12				0.38		0.28
13				3.78		0.03
14				0.25		
15						
16	0.18					
17			0.46			4.01
18	1.60					
19						
20					5.59	0.08
21				0.84		
22		0.03				
23		1.27		0.74		
24	0.20	0.91			0.51	5.23
25		1.50				0.33
26						
27			3.73			
28						
29					0.25	
30				0.53		
31				0.03		
TOTAL	8.94	7.16	4.42	8.51	7.62	14.02

Table B-4. Rainfall data at Knoxville in 1989.

DATE	APRIL	MAY	JUNE	JULY	AUG	SEPT
			(cm)			
1		1.70			0.84	
2		0.89		0.28	4.06	0.64
3	0.58		0.10	0.46		
4	1.27			1.24		
5	1.60	0.56	0.69	0.10		
6		3.94	3.96	0.05	0.18	
7	0.43	0.18	2.44	2.24		
8	0.48			0.15		
9	0.64	0.66	1.91			
10		1.17	0.41			0.08
11						
12		0.13		0.20		
13			3.91	0.81		5.89
14				1.57		4.19
15	0.76	0.08	2.39		1.12	
16		0.03	1.83			
17			1.60	0.36		
18						
19						
20		2.34	1.45	0.97		1.83
21				0.03		2.87
22			0.13	1.73		0.46
23	0.10	0.89	0.99	0.08	3.18	
24				1.19	0.25	2.92
25	0.10					
26					1.42	
27		2.39				1.19
28			0.10			1.63
29	0.46		0.97		0.99	
30	0.89				0.15	
31						
TOTAL	7.31	14.88	22.86	11.46	12.19	21.72

Table B-5. Rainfall data at Milan in 1989.

DATE	APRIL	MAY	JUNE	JULY	AUG	SEPT
	(cm)					
1				0.23		
2		7.75				
3				1.52		
4			0.13			
5	2.82			0.20		
6	1.60		0.66			
7		1.57	0.08			
8		0.10		0.15		
9	0.84					
10	0.28		2.41			1.45
11		0.38		0.03		
12		0.36		1.30		
13			0.43	0.79		0.05
14			1.30	0.28		2.46
15						0.05
16			0.91	1.32		
17		0.64				
18						
19				3.33		
20			1.30		0.05	
21		0.64		1.14		
22				0.43		0.56
23						
24		0.33			0.13	0.18
25		1.70	0.28	0.13		
26						
27						0.15
28			0.03			8.61
29		5.38	1.37			
30			0.08		5.33	
31	0.03			0.48		
TOTAL	6.85	17.57	9.84	10.34	11.91	13.51

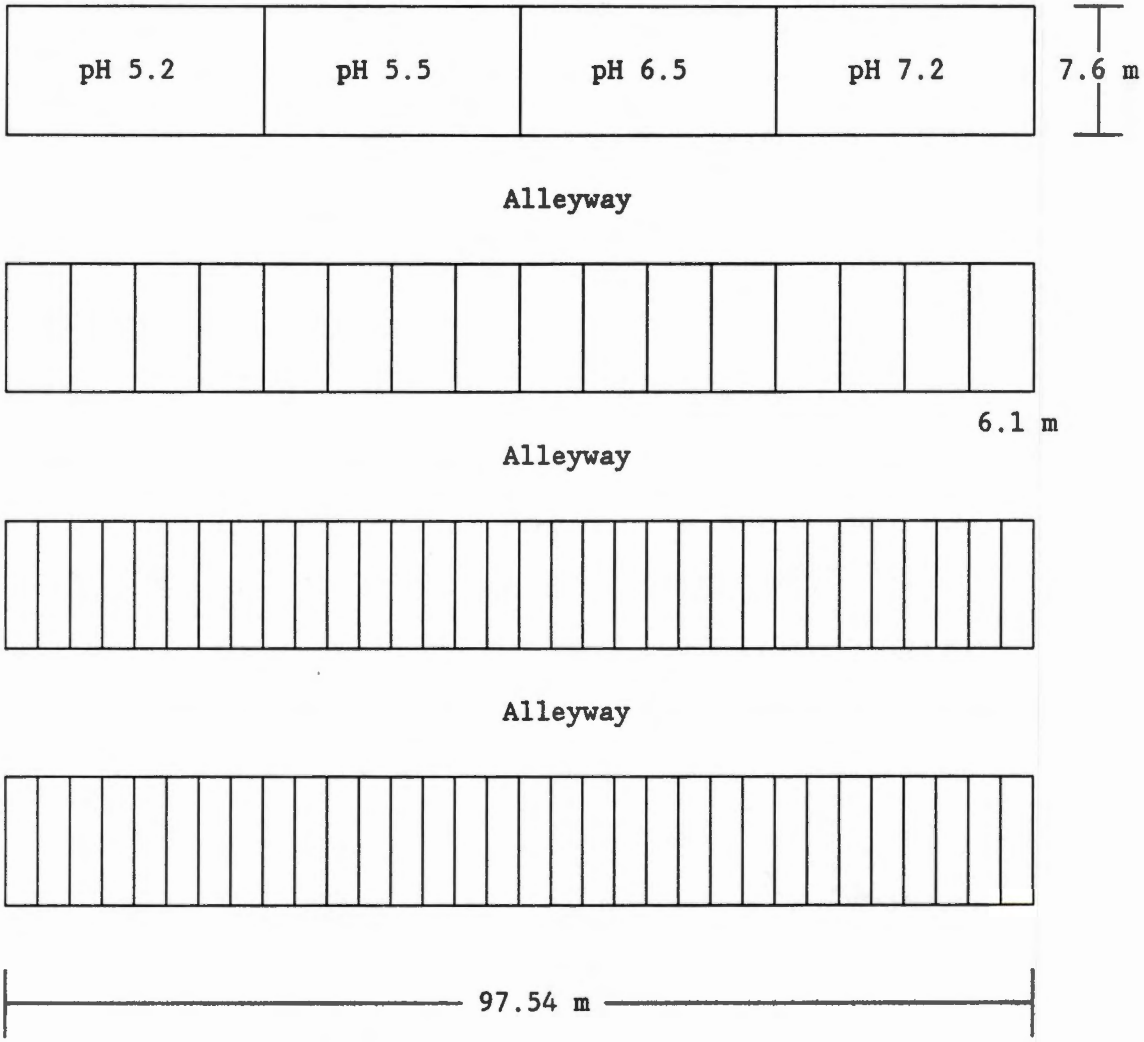
Table B-6. Rainfall data at Springfield in 1989.

DATE	APRIL	MAY	JUNE	JULY	AUG	SEPT
			(cm)			
1		0.23			0.10	
2			0.43	8.08	0.66	0.23
3	1.50			5.79		
4	3.53		4.57	0.56		
5	0.18	2.29	1.07	0.61		
6		1.30	0.61			
7	0.33				2.11	
8	0.48					
9	0.31	0.18	1.70			
10		0.64				2.46
11						0.08
12			0.10	1.02		
13			6.40	1.27		
14			0.10			0.79
15	0.58		4.37			2.29
16			0.40			0.71
17						
18			0.05			
19			3.45	1.58		
20		2.16	0.28	0.31		
21			0.99	0.05		
22						0.13
23		0.56				3.20
24				0.74	1.04	
25					0.33	
26						0.89
27		1.02			0.33	
28			1.09	0.03		
29	0.25		0.08			0.48
30					0.25	1.27
31				0.23	0.20	
TOTAL	7.16	8.38	25.69	20.27	5.02	12.53

APPENDIX C

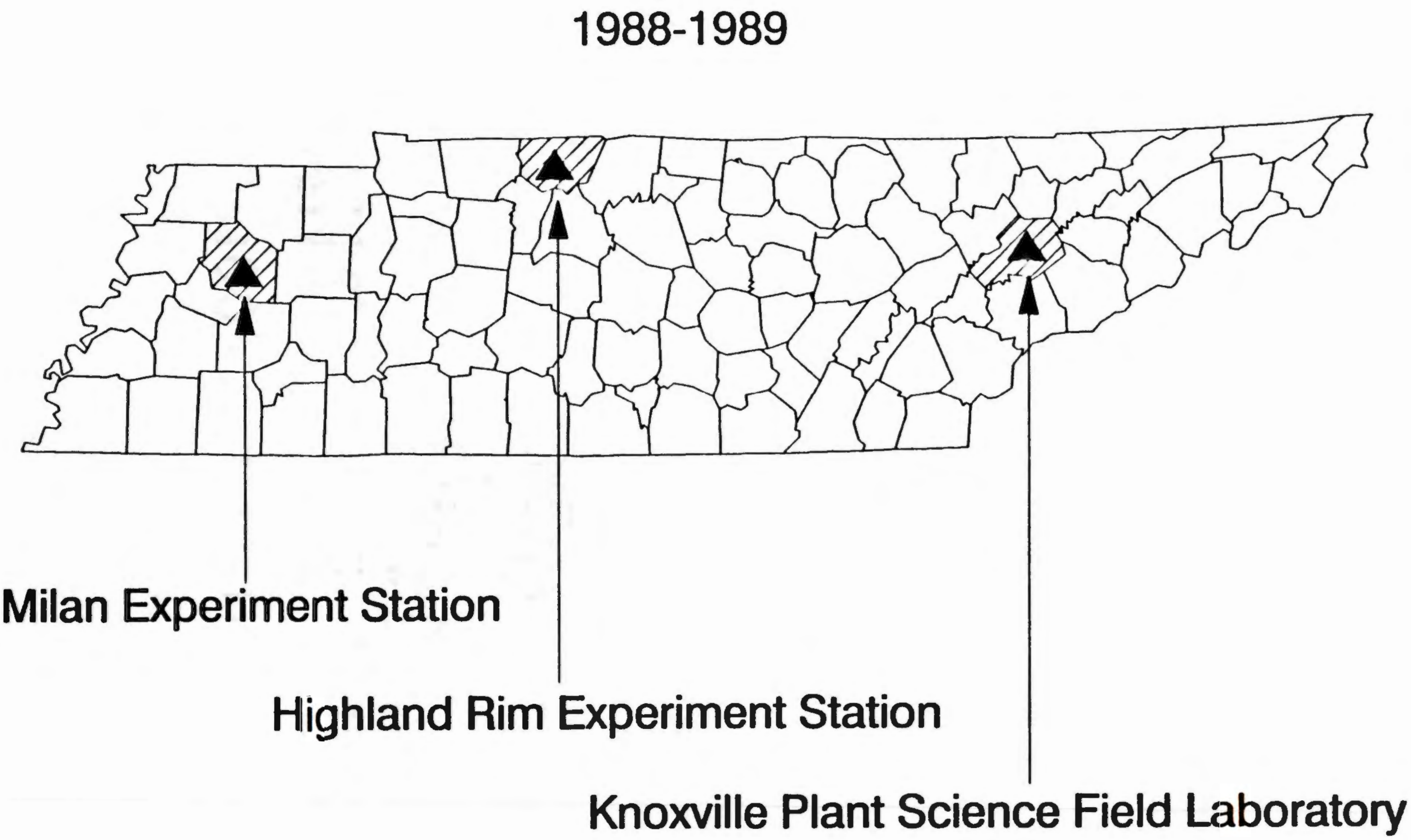
EXPERIMENT LAYOUT AND LOCATION

Field design at all three locations.



Each plot contained 8 rows of soybeans being divided into different years of application. Soybeans were planted on 76.2 cm centers. The alleyway was 6.2 m in length.

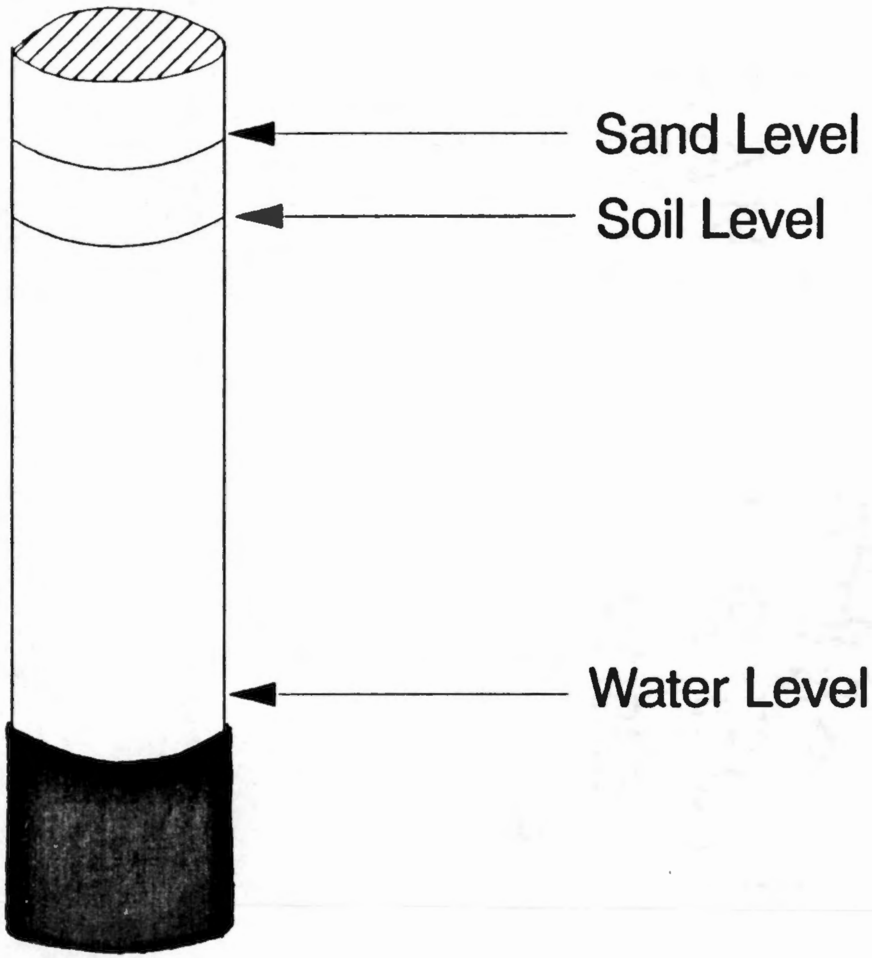
Figure 17. Tennesse map showing experiment locations.



APPENDIX D

BIOASSAY TUBE DIAGRAM

Figure 18. Tube example for greenhouse bioassay.



VITA

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