ADJUVANT EFFECTS ON HERBICIDE ABSORPTION AND TRANSLOCATION

By

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Bispyribac-sodium is an acetolactate synthase (ALS) inhibiting herbicide introduced for control of grasses, broadleaves, and sedges in rice and turf. However, Mississippi and Arkansas rice producers have reported inconsistent barnyardgrass (*Echinochloa crus-galli* Beauv.) control with bispyribac-sodium. Experiments were conducted to determine if inconsistent barnyardgrass control with bispyribac-sodium could be alleviated through adjuvant technology.

Absorption of ^{14}C-bispyribac-sodium was greatest with a proprietary blend of methylated seed oil/organosilicone (MSO/OSL) (0.37 L ha^{-1}) plus urea ammonium nitrate (UAN) (2% v/v) and the proprietary blend of MSO/OSL/UAN (2% v/v) at 80 and 74% of applied ^{14}C-bispyribac-sodium, respectively. The addition of 32% UAN to MSO/OSL and non-ionic organosilicone (OSL/NIS) adjuvant systems resulted in a four- to five-fold increase in absorption compared to treatments without UAN. Maximum absorption was achieved 12 h after application and therefore, bispyribac-sodium should be rainfast at this time. MSO/OSL adjuvants supplied from multiple manufacturers were also

examined to determine which provided the highest levels of ^{14}C-bispyribac-sodium absorption as well as herbicidal efficacy and rainfastness. The addition of Rivet® plus UAN or Sil-MES 100® plus UAN to ^{14}C-bispyribac-sodium resulted in the highest levels of absorption among adjuvants supplied. Bispyribac-sodium applied with no adjuvant or with 32% UAN only resulted in significant reductions in control compared to MSO/OSL adjuvant formulations. Addition of MSO or MSO/OSL adjuvants and UAN to bispyribac-sodium resulted in greater than 85% freshweight reduction of barnyardgrass. Bispyribac-sodium applied with MSO or Dyne-A-Pak® was rainfast by two and four hours after application, respectively. Addition of Sil-MES 100® plus UAN or Inergy® plus UAN to bispyribac-sodium reduced the rainfastness interval to 30 minutes and one hour after herbicide application, respectively. Further experiments were conducted to determine if individual components of 32% UAN as well as ammonium sulfate (AMS) would provide control similar to that of 32% UAN. The addition of ammonium chloride, calcium nitrate, and ammonium sulfate at selected rates provided barnyardgrass control similar to that of 32% UAN.

DEDICATION

I would like to dedicate this research to my beautiful and loving wife Erin and our daughter Rose, you have sacrificed more than anyone to make this possible. You always encouraged me to do my best, even when it meant spending more time at work and less at home. I am looking forward to spending the rest of my life with you. I would also like to dedicate this work to my father and late-mother, Darwin and Dixie Dodds, as well as my stepmother, Donna Dodds. Special thanks are extended to my father- and mother-in-law, Gary and Mardel Robinson. Your support throughout the process of my education has been unwavering.

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TABLE OF CONTENTS

Page

DEDICATION ... ii

ACKNOWLEDGEMENTS ... iii

LIST OF TABLES ... vi

LIST OF FIGURES ... vii

CHAPTER

I. INTRODUCTION ... 1

LITERATURE CITED ... 5

II. EFFECT OF ADJUVANT AND UREA AMMONIUM NITRATE ON BISPYRIBAC-SODIUM ABSORPTION AND TRANSLOCATION ... 8

ABSTRACT ... 8
INTRODUCTION ... 9
MATERIALS AND METHODS ... 11
^{14}C-bispyribac-sodium Adjuvant Study ... 11
^{14}C-bispyribac-sodium Absorption Study ... 15
RESULTS AND DISCUSSION ... 16
^{14}C-bispyribac-sodium Adjuvant Study ... 16
^{14}C-bispyribac-sodium Absorption Study ... 18
LITERATURE CITED ... 22

III. EFFECT OF METHYLATED SEED OIL/ORGANOSILICONE ADJUVANT BLENDS AND UREA AMMONIUM NITRATE ON ABOSRPTION, EFFICACY, AND RAINFASTNESS OF BISPYRIBAC-SODIUM ON BARNYARDGRASS (*ECHINOCHLOA CRUS-GALLI*) ... 31

ABSTRACT ... 31
INTRODUCTION ... 32
MATERIALS AND METHODS ... 35
^{14}C-bispyribac-sodium Adjuvant Study ... 35
Bispyribac-sodium Efficacy Study ... 39

Bispyribac-sodium Rainfastness Study 39
RESULTS AND DISCUSSION 40
^{14}C-bispyribac-sodium Adjuvant Study 40
Bispyribac-sodium Efficacy Study 43
Bispyribac-sodium Rainfastness Study 44
LITERATURE CITED 47

IV. THE EFFECT OF NITROGEN FORM ON EFFICACY OF BISPYRIBAC-SODIUM PLUS A METHYLATED SEED OIL/ORGANOSILICONE ADJUVANT ON BARNYARDGRASS (*ECHINOCHLOA CRUS-GALLI*) 57

ABSTRACT 57
INTRODUCTION 58
MATERIALS AND METHODS 60
RESULTS AND DISCUSSION 63
LITERATURE CITED 66

LIST OF TABLES

2.1. Effect of adjuvant on absorption and translocation of ^{14}C-bispyribac-sodium in barnyardgrass 24

2.2. Effect of time after application on partitioning of ^{14}C-bispyribac-sodium + methylated seed oil/organosilicone (MSO/OSL) adjuvant + urea ammonium nitrate (UAN) in barnyardgrass 26

3.1. Manufacturer and constituents of adjuvants from multiple manufacturers 49

3.2. Effect of adjuvant on absorption and translocation of ^{14}C-bispyribac-sodium in barnyardgrass in experiment one 50

3.3. Effect of adjuvant on absorption and translocation of ^{14}C-bispyribac-sodium in barnyardgrass in experiment two 52

3.4. Efficacy of bispyribac-sodium and methylated seed oil/organosilicone adjuvants from multiple manufacturers on barnyardgrass 54

3.5. Barnyardgrass freshweight reductions using bispyribac-sodium plus a methylated seed oil/organosilicone adjuvant and urea ammonium nitrate 55

4.1. Nitrogen sources and rates used in combination with bispyribac-sodium plus a methylated seed oil/organosilicone adjuvant for control of barnyardgrass 68

4.2. Bispyribac-sodium and methylated seed oil/organosilicone (MSO/OSL) adjuvant plus nitrogen source spray solution pH 69

4.3. Efficacy of bispyribac-sodium and a methylated seed oil/organosilicone adjuvant plus various nitrogen sources 71

LIST OF FIGURES

2.1. Absorption of ^{14}C-bispyribac-Na into barnyardgrass when applied with a methylated seed oil/organosilicone (MSO/OSL) adjuvant + urea ammonium nitrate (UAN) 28

2.2. Distal translocation of ^{14}C-bispyribac-Na in barnyardgrass when applied with a methylated seed oil/organosilicone (MSO/OSL) adjuvant + urea ammonium nitrate (UAN) 29

2.3. Proximal translocation of ^{14}C-bispyribac-Na in barnyardgrass when applied with a methylated seed oil/organosilicone (MSO/OSL) adjuvant + urea ammonium nitrate (UAN) 30

CHAPTER 1

INTRODUCTION

Barnyardgrass [*Echinochloa crus-galli* (L.) Beauv] is a common and competitive weed in rice (*Oryza sativa* L.) (Smith 1968). Smith (1968) reported barnyardgrass densities as low as one plant ft^{-2} reduced rice yields in Arkansas. Barnyardgrass competition with rice for 15 to 20 days reduced rice yields. Barnyardgrass densities of 11 to 269 plants m^{-2} reduced rice yields 25 to 79% with season-long competition. One barnyardgrass plant m^{-2} can reduce yields 65 kg ha^{-1}, whereas 10 plants m^{-2} can reduce yields 10%, and 57 plants m^{-2} can reduce yields 50% (Smith 1988). Barnyardgrass at densities of five to 10 plants m^{-2} is sufficient to warrant control (Smith 1988). Barnyardgrass threshold levels in rice are influenced by fertility, rice cultivar, and rice density. Yield reductions may result from competition for nutrients and light and barnyardgrass should be controlled as soon as possible after emergence (Smith 1968). Propanil is a commonly used herbicide for postemergence control of barnyardgrass in rice (Smith 1965). However, barnyardgrass resistance to propanil has been discovered in the United States (Carey et al. 1995) and around the world (Vasilakoglou et al. 2000). Additionally, barnyardgrass resistance to atrazine and quinclorac has also been reported (Lopez-Martinez et al. 1997)

Bispyribac-sodium is an acetolactate synthase (ALS) inhibiting herbicide for the control of grasses (especially *Echinochloa spp.*), sedges, and broadleaved weeds in rice and turf (Anonymous 2007a; Vencill 2002). ALS-inhibiting herbicides block formation of the branched-chain amino acids leucine, isoleucine, and valine in susceptible species (DeWitt et al. 1999; Shimizu et al. 1994). Although plant death occurs due to events occurring in response to inhibition of ALS, the actual sequence of phytotoxicity is unclear (Vencill 2002).

Bispyribac-sodium may be applied to barnyardgrass from one-leaf to two- to three-tiller stages of growth. Use rates range from 22 to 40 grams ai ha^{-1} and an adjuvant is required (Anonymous 2007b). Williams (1999) reported 98 to 100% barnyardgrass control with 20 to 23 grams ai ha^{-1} of bispyribac-sodium applied mid- to late-postemergence to rice. However, bispyribac-sodium applied to three-tiller barnyardgrass only provided 70% control. Schmidt et al. (1999) reported similar results where 20 to 22 grams ai ha^{-1} provided good control of propanil-resistant barnyardgrass. Webster et al. (1999) reported greater than 90% barnyardgrass control using bispyribac-sodium. However, Mississippi and Arkansas rice producers reported inconsistent barnyardgrass control using bispyribac-sodium (V.F. Carey III, personnel communication). Inconsistent barnyardgrass control may be related to inconsistent use of adjuvant systems with bispyribac-sodium.

The use of selective postemergence herbicides to control annual and perennial grasses has resulted in greater use of crop oils and/or petroleum oil concentrates (Bridges 1989). Adjuvants are used to improve spray delivery, to increase spray retention on weed foliage, and to enhance foliar penetration by postemergence herbicides for increased herbicidal efficacy (Penner 1989). The addition of a spray adjuvant is necessary for maximum bispyribac-sodium efficacy on barnyardgrass (Koger et al. 2007). However, herbicidal efficacy is dependant on the environment, type of herbicide being applied, and the characteristics of the target weed (Bunting et al. 2004; Penner 1989; Roggenbuck et al. 1990).

Hart et al. (1992) found that addition of methylated seed oil (MSO) or DC-X2-5394 increased efficacy and absorption of primisulfuron in giant foxtail (*Setaria faberi* Herrm.) compared to a non-ionic surfactant (NIS). Wills et al. (1998) demonstrated that methylated seed oil/organosilicone adjuvants (MSO/OSL) and crop oil concentrates (COC) applied with imazethapyr provided 24 and 15% greater control, respectively, of barnyardgrass compared to imazethapyr applied with no adjuvant. Wills et al. (1998) also found that the addition of urea ammonium nitrate (UAN) to imazethapyr and adjuvant systems enhanced imazethapyr phytotoxicity. Bunting et al. (2004) observed increased control of giant foxtail when a MSO or MSO plus UAN adjuvant was included with foramsulfuron. Control of giant foxtail with foramsulfuron plus COC or NIS was reduced compared to MSO or NIS adjuvant systems; however, the addition of UAN to COC or NIS increased control of giant foxtail with foramsulfuron compared to COC or NIS alone. Applications of foramsulfuron with MSO or MSO plus UAN increased absorption compared to applications of foramsulfuron plus NIS, COC, NIS plus UAN, or COC plus UAN (Bunting et al. 2004).

Fielding and Stoller (1990a) observed increased velvetleaf (*Abutilon theophrasti* Medik.) control when NIS plus UAN was included with thifensulfuron. Increased absorption of ^{14}C-thifensulfuron in velvetleaf was also observed when NIS or UAN were included. Similarly, chlorimuron with UAN and NIS provided increased velvetleaf control and absorption. Absorption of ^{14}C-chlorimuron was 2%, 21%, 9%, and 32% with the addition of no additive, NIS, UAN, and NIS plus UAN, respectively. (Fielding and Stoller 1990b). Young and Hart (1998) found increased control of giant foxtail when UAN was added to isoxaflutole plus NIS; however, increases in control were not observed with COC and MSO under greenhouse conditions. Bruce et al. (1993) reported increased absorption of radiolabelled nicosulfuron and primisulfuron by quackgrass [*Elytrigia repens* (L.) Nevski] with petroleum oil adjuvants (POA) plus UAN compared to addition of NIS plus UAN or MSO. However, the addition of UAN to NIS increased absorption. Nicosulfuron and primisulfuron plus POA and UAN provided greater quackgrass control than with POA alone. Beckett and Stoller (1991) observed a 2.5-fold increase of ^{14}C-thifensulfuron absorption into velvetleaf when applied with NIS and UAN; however, relative translocation was unaffected by nitrogen additives.

Roggenbuck et al. (1990) observed increased control of velvetleaf with acifluorfen and bentazon when the OSL adjuvant DC-9 was used compared to COC. Boydston and Al-Khatib (1994) indicated that the OSL adjuvant DC-X2-5309 improved control of kochia [*Kochia scoparia* (L.) Schrad.] compared to COC or no adjuvant when applied with bentazon, bromoxynil, pyridate, and terbacil. In the case of bentazon and bromoxynil, the addition of DC-X2-5309 increased uptake four- to five-fold and two-fold, respectively, compared to bentazon or bromoxynil applied alone. Reddy and Singh (1992) observed increased velvetleaf control when glyphosate was applied with an organosilicone/non-ionic surfactant (OSL/NIS) system compared to a COC adjuvant. Roggenbuck et al. (1993) found that Sylgard 309® improved control of velvetleaf by glyphosate compared to other OSL and oil based adjuvant systems. The addition of UAN to Sylgard 309® increased control of velvetleaf and giant foxtail with imazethapyr compared to applications utilizing Sun-It II® or Sun-It II® plus UAN. Imazethapyr applied with Sun-It II® plus UAN increased control of velvetleaf compared to similar applications without UAN. In addition, absorption of ^{14}C-bentazon and ^{14}C-acifluorfen were increased when applied with Sylgard 309® compared to similar applications utilizing COC adjuvants.

Research indicates that adjuvants differ in their effectiveness (Nalewaja et al. 1991) and identification of the appropriate adjuvant for a specific herbicide use is important to obtain maximum herbicidal activity (Nalewaja et al. 1995; Wanamarta et al. (1989). Therefore, this research was conducted to determine which adjuvants optimized the activity of bispyribac-sodium. Initial experiments examined the effect of various adjuvant systems and additives including: MSO/OSL, OSL/NIS, MSO/OSL plus UAN, OSL/NIS plus UAN, OSL/NIS plus N, and MSO/OSL/UAN on absorption and translocation of ^{14}C-bispyribac-sodium. Further experiments were conducted to determine the effects of MSO/OSL adjuvants supplied from multiple manufacturers on absorption, efficacy, and rainfastness of bispyribac-sodium. Experiments were also conducted to quantify the effects of nitrogen form on bispyribac-sodium plus MSO/OSL efficacy.

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CHAPTER II

EFFECT OF ADJUVANT AND UREA AMMONIUM NITRATE ON BISPYRIBAC-SODIUM ABSORPTION AND TRANSLOCATION

ABSTRACT

Inconsistent control of barnyardgrass with bispyribac-sodium may be alleviated through adjuvant technology. Experiments were conducted to determine the effect of adjuvant, urea ammonium nitrate (UAN), and time after treatment on absorption and translocation of bispyribac-sodium in barnyardgrass. Additional experiments were conducted to determine when maximum absorption and translocation occurred using a methylated seed oil/organosilicone adjuvant (MSO/OSL) plus UAN (0.37 L ha^{-1} and 2% v/v). In the initial experiment, ^{14}C-bispyribac-treated leaves, non-treated treated leaves, and roots were collected 6 and 24 h after application. Recovery of radiolabelled material was greater than 94%. Absorption was greatest with tankmixed MSO/OSL (0.37 L ha^{-1}) plus UAN (2% v/v) and the proprietary blend of MSO/OSL/UAN (2% v/v) at 80 and 74% of applied ^{14}C-bispyribac-sodium, respectively. Translocation to non-treated leaves and roots was also highest with these treatments. All other adjuvant systems provided 47% or less absorption. Increased translocation appears to be due to greater herbicide absorption, not an increase in translocation rate. However, the addition of 32% UAN to MSO/OSL and non-ionic organosilicone (OSL/NIS) adjuvant systems resulted in a four- to five-fold increase in absorption compared to treatments without UAN. Recovery of ^{14}C-bispyribac-sodium in additional experiments generally decreased as time after

application increased; however, recovery was 86% or greater for all time intervals. By 12 h after application, 68% of applied ^{14}C-bispyribac-sodium was absorbed. At this time, ^{14}C-bispyribac-sodium was partitioned within the plant in the following manner: 48% in the treated area, 10% in leaf tissue from treated area to tip of the treated leaf, 1.9% in leaf tissue from treated area to collar region of the treated leaf, 1.6% in remaining leaves from collar of treated leaf upward, 5.3% in remaining leaves from collar of treated leaf downward to soil line, and 0.7% in the roots. These data indicate that maximum absorption was achieved with a tankmix of MSO/OSL and UAN or the MSO/OSL/UAN blend. Maximum absorption was achieved 12 h after application and therefore, bispyribac-sodium should be rainfast at this time.

Nomenclature: bispyribac-sodium; barnyardgrass, *Echinochloa crus-galli* (L.) Beauv. ECHCG.

Abbreviations: UAN, urea ammonium nitrate; MSO/OSL, methylated seed oil/organosilicone adjuvant; OSL/NIS, non-ionic organosilicone adjuvant; h, hour; L ha^{-1}, liters per hectare;

INTRODUCTION

Barnyardgrass [*Echinochloa crus-galli* (L.) Beauv.] is one of the most troublesome and competitive annual weeds in rice (*Oryza sativa* L.) (Smith 1968). Single-year economic threshold values range from 2.86 to 3.01 barnyardgrass plants m^{-1} (Lindquist and Kropff 1996). Barnyardgrass densities greater than five plants m^{2} warrant control (Smith 1988). Optimum yield and economic return in rice is achieved if competition between rice and barnyardgrass is eliminated prior to the weed reaching jointing stage and/or the crop reaching tillering stage (Smith 1974).

Bispyribac-sodium is an acetolactate synthase (ALS) inhibiting herbicide recently introduced to control grasses, sedges, and broadleaf weeds when applied postemergence in rice (DeWitt et al. 1999; Vencill 2002). Bispyribac-sodium can be applied to barnyardgrass from second leaf to tillering (Anonymous 2006). However, inconsistent barnyardgrass control with bispyribac-sodium has become a problem for Mississippi and Arkansas rice producers (V.F. Carey III, personal communication). Erratic control may be related to inconsistent use of adjuvant systems with bispyribac-sodium.

The use of selective postemergence herbicides to control annual and perennial grasses has resulted in greater use of crop oils and/or petroleum oil concentrates (Bridges 1989). However, the type of adjuvant used can affect control by altering herbicide absorption. Differences in barnyardgrass control using crop oil concentrate (COC), MSO, and OSL adjuvants with propanil have been observed (Jordan et al. 1997). The absorption of ^{14}C-thifensulfuron by velvetleaf (*Abutilon theophrasti* Medik.) increased 2.5-fold with the addition of UAN to the spray solution (Beckett and Stoller 1991). Using urea-based foliar nitrogen and OSL adjuvants increased burndown control of bristly starbur (*Acanthospermum hispidum* DC.) and Florida beggarweed [*Desmodium tortuosum* (Sw.) DC] compared to non-ionic surfactants (Hammes 1993). Adding UAN to non-ionic surfactants or petroleum oil adjuvants increased foliar absorption of ^{14}C-nicosulfuron and ^{14}C-primisulfuron (Bruce et al. 1993). Research indicates that adjuvants differ in their effectiveness (Nalewaja et al. 1991), and that identification of the appropriate adjuvant for a specific herbicide use is required to maximize herbicidal activity (Wanamarta et al. 1989). Therefore, the objectives of this research were to (1) determine the effect of adjuvant and UAN on absorption and translocation of bispyribac-sodium by barnyardgrass, and (2) using the optimum adjuvant system identified in objective one, determine the time required for maximum absorption and translocation of bispyribac-sodium by barnyardgrass.

MATERIALS AND METHODS

^{14}C-bispyribac-sodium Adjuvant Study

Barnyardgrass seed were planted approximately 2.5-cm deep in 40 cm^3 plastic conetainers[1] containing a 1:1 mixture of Leeper silty clay loam (fine, montmorillonitic nonacid, thermic Chromudertic Haplaquept) soil and peat[2], and conetainers were sub-irrigated with tap water for the duration of the study. Plants were thinned to 1 plant per conetainer within 1 wk of emergence, and grown at 35/30°C day/night temperature. Natural light was supplemented with light from sodium vapor lamps[3] to provide a 16-hour photoperiod. Approximately 1.5 wk after thinning (8-cm tall plants), all plants were fertilized[4] with 168, 56, and 56 kg ha^{-1} (actual nitrogen, phosphorus, and potassium, respectively). The fertilization rate was based on soil test recommendations for rice production in Mississippi.

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[1] D40 (40 cm^3) Deepot Cell, Stuewe and Sons, Inc., 2290 SE Kiger Island Dr., Corvallis, OR 97333-9425.

[2] Baccto Sphagnum Peat Moss, Michigan Peat Co., P.O. Box 980129, Houston, TX 77098-0129.

[3] General Electric Sodium Vapor Lamps, Lucalox LU 400, General Electric Consumer and Industrial Lighting, 1975 Noble Rd., Nela Park, Cleveland, OH 44112.

[4] Expert Gardener, Azalea, Camellia, and Rhododendron Water Soluble Plant Food, 30-10-10, Chemsico, a Division of United Industries Corp., P.O. Box 142642, St. Louis, MO 63114-0642.

Approximately 1.5 weeks after fertilizing, plants with a fully expanded second true leaf were selected for treatment (15 to 20-cm in height). Approximately two to four cm from the collar, 2.5-cm of the second true leaf of each plant was covered with adhesive backed paper[5] prior to pre-spraying. Plants were pre-sprayed with non-radiolabelled bispyribac-sodium[6] at 22.5 g ai ha^{-1} plus the following adjuvants: (1) OSL/NIS[7] at 0.25% v/v; (2) OSL/NIS + 32% UAN at 0.25 and 2% respectively; (3) OSL/NIS and + Coron (N) 25-0-0[8] at 0.25%v/v and 2.34 L ha^{-1} respectively, (4) MSO/OSL[9] at 0.37 L ha^{-1}, (5) MSO/OSL + 32% UAN at 0.37 L ha^{-1} and 2% respectively, (6) a proprietary blend of MSO/OSL/UAN[10] at 2% v/v. A bispyribac-sodium with no adjuvant treatment was also included for comparison purposes. All plants were pre-sprayed using a compressed air spray chamber with an XR110015E[11] flat fan nozzle at an application volume of 140 L

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[5] Post-It™ Brand Fax Transmittal Memo 7671, 3M Commercial Office Supply Division, 3M Center, St. Paul, MN 55144-1000.

[6] Regiment 80 WP; Valent U.S.A Corp., P.O. Box 8025, Walnut Creek, CA 94956-8025.

[7] Kinetic HV®, 99% blend of polyalkyleneoxide modified polydimethylsiloxane and polyoxypropylene-polyoxyethylene copolymers. Helena Chemical Co., Suite 300, 225 Schilling Blvd., Collierville, TN 38017.

[8] CoRoN 25-0-0®, Controlled Release Nitrogen Fertilizer, 18.8% Urea Nitrogen, 6.2% Water Soluble Nitrogen, Helena Chemical Co., Suite 300, 225 Schilling Blvd., Collierville, TN 38017.

[9] Dyne-Amic®, 99% blend of polyalkyleneoxide modified polyimethylsiloxane, nonionic emulsifiers, and methylated vegetable oils, Helena Chemical Co., Suite 300, 225 Schilling Blvd., Collierville, TN 38017.

[10] Dyne-A-Pak®, blend of proprietary blend of polyalkyleneoxide modified polyimethylsiloxane, nonionic emulsifiers, methylated vegetable oils, and nitrogen fertilizer solution, Helena Chemical Co., Suite 300, 225 Schilling Blvd., Collierville, TN 38017.

[11] TeeJet, PO Box 7900, Wheaton, IL 60189-7900.

ha^{-1} and a pressure of 240 kPa. Immediately after pre-spraying plants, ^{14}C-bispyribac-sodium plus adjuvant was applied to the area covered during pre-spraying. The ^{14}C solution was prepared by dissolving ^{14}C-bispyribac-sodium [^{14}C-benzene labeled with 1.2 GBq mmol^{-1} specific activity, 94.5% radio-chemical purity] in an aqueous solution of a commercial formulation of bispyribac-sodium[6] and selected adjuvant systems to give a final concentration of 0.16 g ai L^{-1}. The radiochemical purity of the ^{14}C-bispyribac-sodium was verified using reversed-phase high performance liquid chromatography[12] and liquid scintillation counting techniques[13]. A 10-μl volume of the final ^{14}C-bispyribac-sodium and adjuvant solution containing 4.31 kBq was placed on the adaxial surface of the second fully expanded true leaf using a 10-μl pipette[14]. To ensure that the applied ^{14}C-herbicide solution remained in the treatment area, the treated leaf was held in a horizontal position using chenille stems, and lanolin barriers were placed traverse to each edge of the treated leaf zones.

Plants were harvested 6 h and 24 h after treatment with ^{14}C-bispyribac-sodium. The treated portion of the second true leaf was excised, and ^{14}C-bispyribac-sodium remaining on the leaf surface was removed by washing in 10-ml deionized water for 15 s. Next, the treated portion of the leaf was washed in 10-ml chloroform[15] for 15 s in

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[12] Hewlett Packard Series 1100 High Performance Liquid Chromatograph equipped with Chemstation software, an auto-injector, and photodiode array detector. 3000 Hanover St., Palo Alto, CA 94304-1185.

[13] Packard 1900 CA TriCarb Liquid Scintillation Counter, PerkinElmer Life and Analytical Sciences, 710 Bridgeport Ave., Sheldon, CT 06484.

[14] Gilson Pipetman, Gilson, Inc., 3000 Parmenter, P.O. Box 620027, Middleton, WI 53562.

[15] Chloroform, CX 1058-1 HPLC Grade, EM Science, 480 S. Democrat Rd, Gibbstown, NJ 08027.

order to remove ^{14}C-bispyribac-sodium from the epicuticular wax. Plants were sectioned into the treated portion of the leaf, all other leaves, and roots. After washing, the treated portion of each leaf was placed in a glass scintillation vial[16] and lyophilized[17]. Remaining plant fractions were placed in paper coin envelopes and lyophilized. A 1-ml aliquot was withdrawn from each rinsate and mixed with 10 ml of liquid scintillation cocktail[18] for quantification by liquid scintillation spectrometry. Plant samples were combusted[19], and the evolved $^{14}CO_2$ trapped in 10-ml liquid scintillation cocktail[20]. Radioactivity (dpm) in leaf washes and oxidations were quantified using liquid scintillation spectrometry[13] using internal calibration and automatic quench correction.

The sum of ^{14}C located in the leaf washes and each plant fraction was considered amount of ^{14}C-recovered. The amount of radioactivity located in the plant fractions was considered absorbed and was expressed as percent of applied radioactivity. Radioactivity located in plant fractions other than the treated leaf was considered translocated and was expressed as percent of applied.

Treatments were arranged in a two-factor factorial arrangement in a randomized complete block design: factor A was time after application and factor B was adjuvant

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[16] Disposable Glass Scintillation Vial; Wheaton Science Products, 1501 N. 10th St., Millville, NJ 08332-2093.

[17] Freeze Dryer, VisTis Research Equipment, Gardiner, NY 12525.

[18] Ultima Gold Universal LSC-Cocktail, PerkinElmer Life and Analytical Sciences, 549 Albany St., Boston, MA 02118-2513.

[19] OX-500 Biological Oxidizer, R.J. Harvey Instrument Company, 123 Patterson St., Hillsdale, NJ 07642.

[20] Carbon-14 Cocktail, R.J. Harvey Instrument Company, 123 Patterson St., Hillsdale, NJ 07642.

system. Six replications for each treatment were used in each study, and the study was repeated at a different time. Where no significant time by adjuvant interaction was observed, data collected 6 h and 24 h after application were pooled for statistical analysis. Data were also pooled across experiment due to no interactions. Data were subjected to combined analysis of variance (ANOVA), and means were separated using Fisher's protected least significance difference (LSD) test at the 5% level of probability (Culpepper et al. 1999).

^{14}C-bispyribac-sodium Absorption Study

Barnyardgrass plants were grown, fertilized, and treated with non-radiolabelled and radiolabelled material in the same manner as described previously. The only adjuvant solution evaluated in this study was MSO/OSL + 32% UAN at 0.37 L ha^{-1} and 2% v/v respectively. The adjuvant treatment was selected based on absorption levels in the above study. Plants were treated with ^{14}C-bispyribac-sodium + MSO/OSL + UAN to determine the minimum time requirement for maximum absorption and translocation. The following time periods were evaluated: 0.5, 1, 2, 3, 6, 12, 24, 48, and 96 hours after application. Plants were divided into the following fractions to determine translocation patterns: treated area, leaf tissue from treated area to leaf tip, leaf tissue from treated area to collar of the treated leaf, plant material from collar of treated leaf to plant tip, plant material from soil line to collar of treated leaf, and roots. The ^{14}C-bispyribac-sodium solution was prepared, applied and radioactivity was quantified in each leaf wash and respective plant fraction in the same manner as described previously. Radioactivity located in plant fractions was considered absorbed and was expressed as percent of applied radioactivity. Radioactivity located in plant fractions other than the treated leaf was considered translocated and was expressed as percent of applied radioactivity.

Distal translocation was calculated as translocation of radiolabelled material in leaf tissue from the treated area to the leaf tip of the treated leaf and expressed as percent of applied radioactivity. Proximal translocation was calculated by combining amounts of radioactivity found in the tissue other than the treated area or the area on the treated leaf distal to the treated area and was expressed as percent of applied radioactivity. Treatments were applied to plants arranged in a randomized complete block design. Each treatment was replicated six times, and the experiment was repeated. Data were pooled across experiment due to no treatment by experiment interaction. Data were subjected to combined analysis of variance (ANOVA) and means were separated using Fisher's protected least significance difference (LSD) test at the 5% level of probability. Absorption and translocation were modeled over time. Logarithmic, quadratic, and inverse modeling was attempted; however, all resulted in r-square values <0.56 due to the nonlinear response. Non-linear regression was used to fit a bi-phasic model to absorption, and distal and proximal translocation data.

RESULTS AND DISCUSSION

^{14}C-bispyribac-sodium Adjuvant Study

Recovery levels ranged from 94 to100%. The general trend was to have lower recovery where more of the herbicide was absorbed. Lower recovery may be due to incomplete recovery of all root fibers which may have contained ^{14}C or exudation of ^{14}C into the soil column in which the plants were growing. The majority of the ^{14}C-bispyribac recovered in the leaf washes was in the initial water rinse (Table 2.1). These results are similar to those of Beckett and Stoller (1991). The highest levels of non-absorbed ^{14}C were obtained with no adjuvant, MSO/OSL, OSL/NIS, and OSL/NIS + N, with 88, 70, 87, and 80% of applied ^{14}C not absorbed, respectively. No significant differences in recovery of ^{14}C were observed in the chloroform wash which ranged from 2 to 8%. Chloroform washes were clear, indicating that the ^{14}C did not come from ruptured epidermal cells, but from the cuticle (Beckett and Stoller 1991). Addition of an OSL/NIS or MSO/OSL adjuvant resulted in 9 and 18% absorption respectively, compared to 4% absorption with no adjuvant (Table 2.1). The inclusion of UAN with OSL/NIS or MSO/OSL adjuvants increased absorption four- to five-fold to 47 and 80%, respectively, compared to OSL/NIS or MSO/OSL alone. Results are similar to those of Beckett and Stoller (1991) who observed approximately a 2.5 fold increase in absorption of ^{14}C-thifensulfuron with the addition of UAN in velvetleaf. Improved absorption of primisulfuron and nicosulfuron in quackgrass (*Elytrigia repens* (L.) Desv. ex B.D. Jackson) using UAN has also been observed (Bruce et al. 1993). The proprietary MSO/OSL/UAN blend adjuvant provided 74% absorption, which was similar to that of MSO/OSL + UAN tankmixed. Tankmixing OSL/NIS + N did not provide a significant increase in absorption (16%) compared to OSL/NIS (9%) or no adjuvant (4%). Increases in absorption with MSO/OSL compared to OSL adjuvants with ^{14}C-clethodim have been observed (Culpepper et al. 1999); however, these data did not indicate that this occurred with ^{14}C-bispyribac-sodium.

Regardless of adjuvant system, at least 85% of the absorbed ^{14}C remained in the treated leaf. Translocation of ^{14}C to other leaves and/or roots ranged from 9 to 15%. Based on these data, the increase in the amount of ^{14}C located in the non-treated leaves and roots with adjuvant systems using UAN may be an artifact of increased absorption of ^{14}C, not an increase in the rate of translocation (Beckett and Stoller 1991). Bispyribac-sodium applied with MSO/OSL + UAN or MSO/OSL/UAN resulted in the highest absorption and translocation. Increased absorption and translocation due to the addition of UAN to MSO/OSL or a proprietary blend of MSO/OSL/UAN corresponds to greater bispyribac efficacy on barnyardgrass when compared to bispyribac applied with surfactant but without UAN (Koger et al. 2007).

^{14}C-bispyribac-sodium Absorption Study

The amount of ^{14}C located in the leaf washes and each plant fraction was summed and considered ^{14}C-recovered which ranged from 86 to 100%. As time after application increased, ^{14}C recovery levels decreased. Significant reductions in recovery of ^{14}C were observed 48 and 96 h after application (Table 2.2). Decreased recovery 48 and 96 h after treatment was most likely due to more radiolabelled material being translocated into the roots and/or incomplete recovery of fine root fibers from the soil column or exudation of some ^{14}C into the soil column in which the plants were growing. Absorption of ^{14}C increased dramatically with time after application eventually peaking at 10 h after application with 63% (± 5%) applied ^{14}C-bispyribac-sodium + MSO/OSL + UAN absorbed (Fig. 2.1). Decreased absorption at 48 and 96 h after treatment may be related to lower recovery of radiolabelled material. Accordingly, at 12 h after application, ^{14}C-bispyribac-sodium + MSO/OSL + UAN found in water and chloroform washes were minimal (Table 2.2). Radioactivity measured in the water wash was greatest 0.5 h after application and declined steadily as time after application increased until 6 h after application. No differences in the level of radioactivity located in the chloroform wash were observed regardless of time interval. Radioactivity in the chloroform wash ranged from 6 to 12% of applied material, indicating that little radiolabelled material was located in the epicuticular wax (Table 2.2).

Highest levels of radioactivity in the plant were found in the treated leaf area (42 to 51% of applied radioactivity), from 1 to 12 h after application (Table 2.2). Prior to 1 h after application, significant amounts of radiolabelled material were removed during the water and chloroform washes corresponding to reduced levels of radioactivity in the treated area. Beyond 12 h after application, significant amounts of radioactivity were translocated (Table 2.2). Translocation of radiolabelled material out of the treated area beyond 12 h after application resulted in significant decreases in the amount of herbicide located in the treated area compared to time periods prior to 12 h after application.

Distal translocation was greatest 60 h after application with 22% (± 2%) of the applied radiolabelled material partitioned in leaf tissue distal the treated area of the treated leaf (Figure 2.2). Distal translocation increased steadily until 60 h after application after which time translocation stabilized. Highest levels of radioactivity in the entire leaf (containing the treated area) were found 3 h after application at 53% of applied material (data not shown).

Maximum proximal translocation of 11% (± 1%) was observed 35 h after application (Figure 2.3). Similar to distal translocation, proximal translocation increased steadily and eventually stabilized as time after application increased. Examining each plant fraction individually, translocation of radiolabelled material in leaf tissue from treated area to collar was maximized 12 h after application (Table 2.2). Translocation of radiolabelled bispyribac-sodium + MSO/OSL + UAN to plant material from collar of the treated leaf to soil line was maximized 12 h after application. Translocation from the collar of the treated leaf to remaining aerial portions of the plant was greatest 24 h after application. About 1.9% of the applied radioactivity was located in this region 24 h after application. Radioactivity located in the roots of barnyardgrass did not increase significantly beyond 24 h after application.

In conclusion, adjuvant systems that provided the highest absorption and translocation of bispyribac-sodium were a tankmix of MSO/OSL + UAN and the proprietary blend MSO/OSL/UAN. The addition of UAN to all adjuvant systems resulted in a four- to five-fold increase in the amount of absorption compared to adjuvant systems without UAN. Using the MSO/OSL + UAN adjuvant system with bispyribac-sodium resulted in the highest levels of absorption 10 h after application. Based on the amount of applied radioactivity translocated distally and proximally, distal translocation of bispyribac-sodium + MSO/OSL + UAN appears to be favored over proximal translocation (Beckett and Stoller 1991). Based on absorption levels after application, the time period required between bispyribac-sodium application and a rainfall event corresponding to optimal herbicide efficacy should be decreased when UAN is added to the bispyribac-sodium + adjuvant solution. Koger et al. (2007) reported applying UAN and an OSL/NIS or MSO/OSL surfactant with bispyribac-sodium enhanced efficacy and reduced the time period required between bispyribac-sodium application and a washoff rainfall event. Their addition of UAN decreased the rainfast period from 8 h (registered rainfast period) to 1 to 4 h (99 to 100% control) when either an OSL/NIS or MSO/OSL surfactant were applied with bispyribac-sodium, respectively.

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(*Echinochloa crus-galli*). II. Absorption and Translocation. Weed Science 55(5): 406-411. Weed Science Society of America, Allen Press Publishing.

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Table 2.1. Effect of adjuvant on absorption and translocation of ^{14}C-bispyribac-sodium in barnyardgrass.[a]

	^{14}C in leaf wash		^{14}C in plant fractions[b]					
					Roots[c]			
				Remaining	Time (h)			Relative
Adjuvant	Water	Chloroform	Treated leaf	leaves	6	24	Absorption	translocation[d]
	---% of applied---							
None	88	8	3	0.6	0.03	0.06	4	12
OSL/NIS[e]	87	2	8	0.8	0.05	0.19	9	10
OSL/NIS + UAN[e]	44	2	43	3.4	0.22	0.61	47	9
OSL/NIS + N[e]	80	2	14	1.6	0.09	0.28	16	15
MSO/OSL[e]	70	7	16	1.6	0.07	0.28	18	10
MSO/OSL + UAN[e]	12	3	72	6.7	0.43	1.16	80	10

Table 2.1 (continued) Contd.

MSO/OSL/UAN[e]	19	3	63	9.9	0.68	1.97	74	15
LSD (0.05)	13	NS	15	1.6	0.31		14	NS

[a] Plants were grown in the greenhouse prior to and after ^{14}C-bispyribac-sodium application. Greenhouse was maintained at 35/30 C day/night temperature with a 16-h photoperiod.

[b] ^{14}C-bispyribac-sodium distribution throughout plant is based on percent of ^{14}C-bispyribac-sodium absorbed averaged over 6 and 24 h after treatment.

[c] Results separated by time after application (indicated as 6 and 24) due to significant ($p \leq 0.05$) time by adjuvant interaction.

[d] The amount of ^{14}C translocated out of the treated leaf as a percentage of total ^{14}C absorption.

[e] Abbreviations: non-ionic organosilicone adjuvant (OSL/NIS), urea ammonium nitrate (UAN), nitrogen (N), methylated seed oil/organosilicone adjuvant (MSO/OSL), proprietary blend of methylated seed oil/organosilicone adjuvant and urea ammonium nitrate (MSO/OSL/UAN).

Table 2.2. Effect of time after application on partitioning of ^{14}C-bispyribac-sodium + methylated seed oil/organosilicone (MSO/OSL) adjuvant + urea ammonium nitrate (UAN) in barnyardgrass.[a]

Time after application (h)	^{14}C in leaf wash		^{14}C in plant fractions						Recovery
	Water	Chloroform	Treated area	Leaf tissue from treated area to leaf tip	Leaf tissue from treated area to collar	Plant material from collar of treated leaf to plant tip	Plant material from soil line to collar of treated leaf	Roots	
	% of applied								
0.5	70	6	24	0	0.1	0.0	0.0	0.1	100
1	47	9	42	1	0.1	0.0	0.1	0.1	99
2	41	8	44	4	0.2	0.1	0.8	0.3	98
3	35	8	51	2	0.5	0.2	0.9	0.4	98
6	26	12	45	7	0.9	0.8	2.6	0.7	95
12	19	6	48	10	1.9	1.6	5.3	0.7	93

Table 2.2 (continued) Contd.

24	29	6	33	17	2.0	1.9	4.9	1.5	95
48	24	5	26	22	1.6	1.9	5.4	1.8	88
96	26	5	24	20	1.2	2.0	4.6	2.0	86
LSD (0.05)	15	NS	9	3	1.1	0.4	2.6	0.6	5

[a] Plants were grown in the greenhouse prior to and after ^{14}C-bispyribac-sodium application. Greenhouse was maintained at 35/30 C day/night temperature, with a 16-h photoperiod.

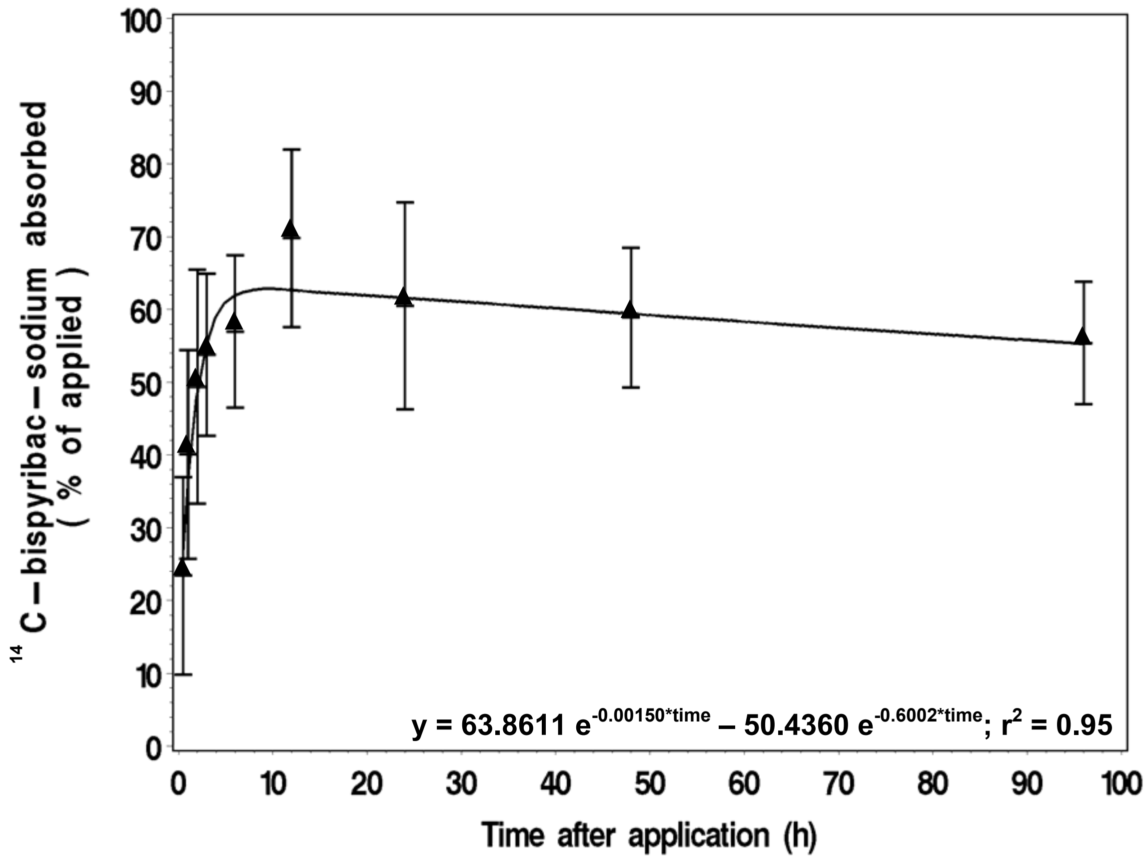

Figure 2.1. Absorption of ^{14}C-bispyribac-Na into barnyardgrass when applied with a methylated seed oil/organosilicone (MSO/OSL) adjuvant + urea ammonium nitrate (UAN).

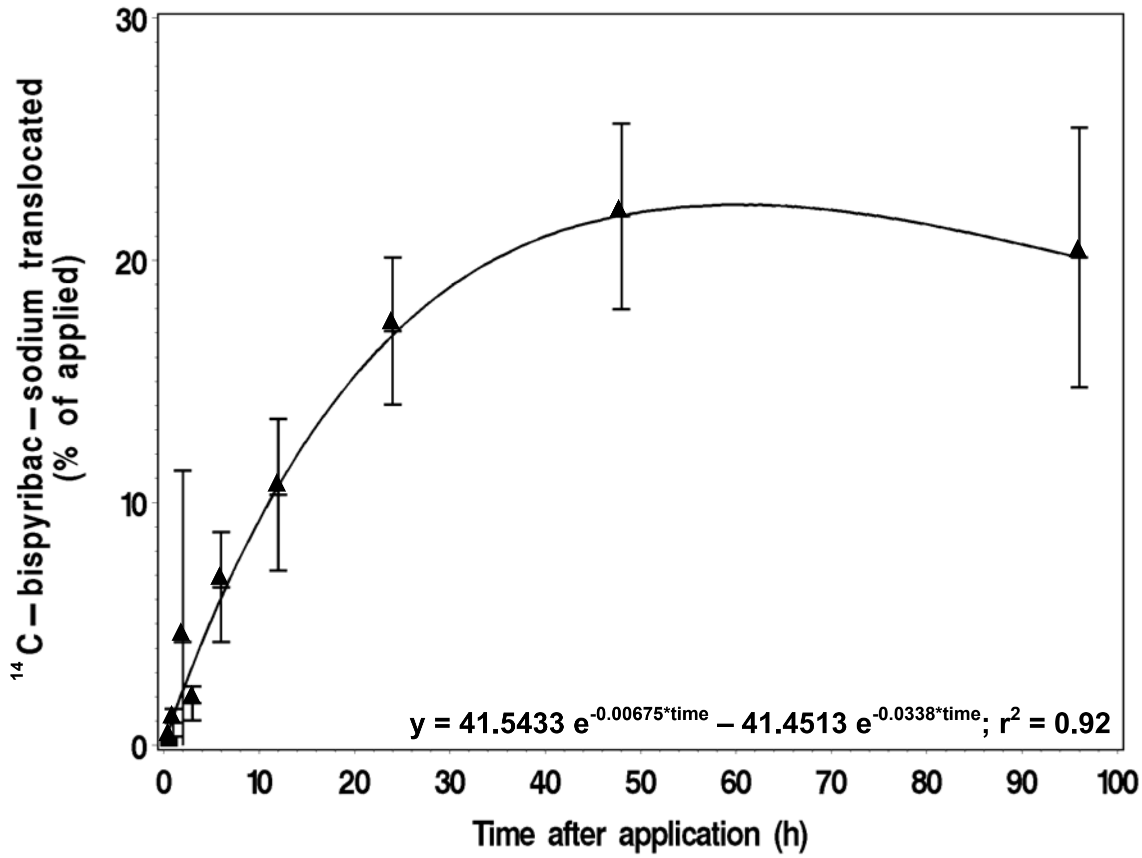

Figure 2.2. Distal translocation of ^{14}C-bispyribac-Na in barnyardgrass when applied with a methylated seed oil/organosilicone (MSO/OSL) adjuvant + urea ammonium nitrate (UAN).

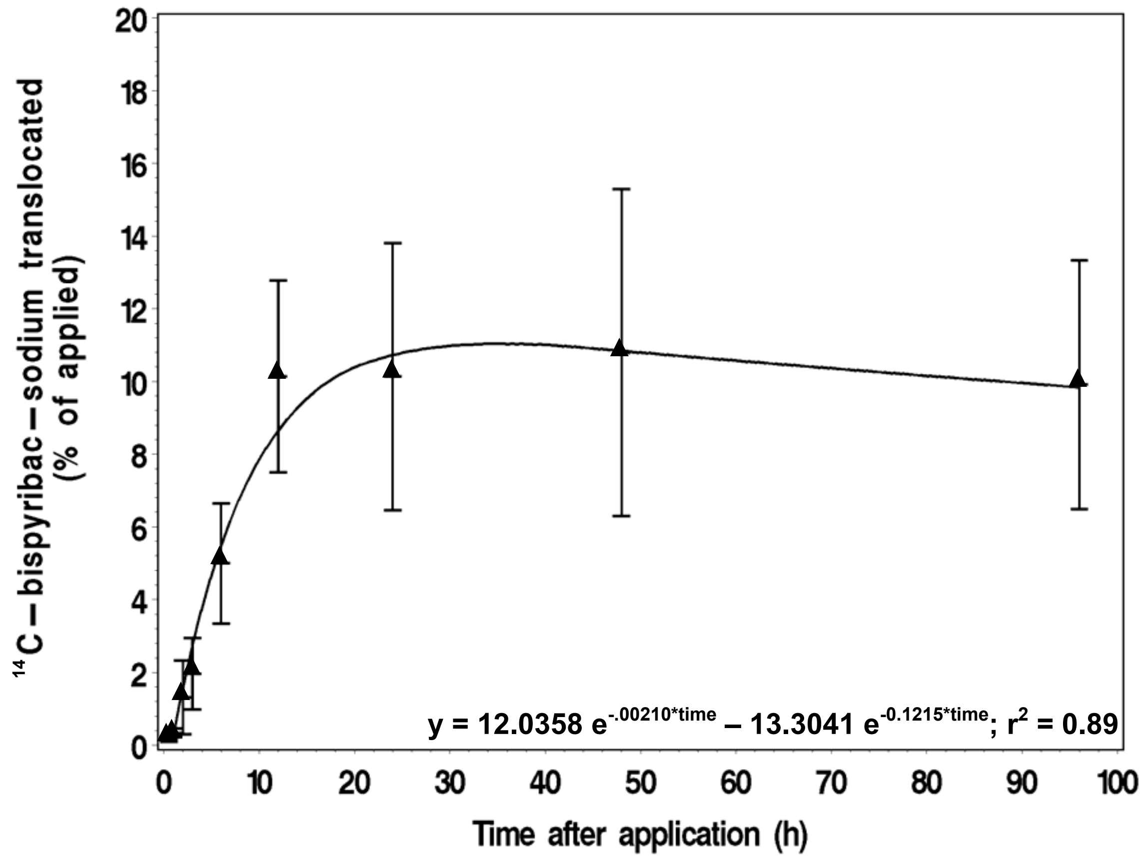

Figure 2.3. Proximal translocation of ^{14}C-bispyribac-Na in barnyardgrass when applied with a methylated seed oil/organosilicone (MSO/OSL) adjuvant + urea ammonium nitrate (UAN).

CHAPTER III

EFFECT OF METHYLATED SEED OIL/ORGANOSILICONE ADJUVANT BLENDS AND UREA AMMONIUM NITRATE ON ABSORPTION, EFFICACY, AND RAINFASTNESS OF BISPYRIBAC-SODIUM ON BARNYARDGRASS (*ECHINOCHLOA CRUS-GALLI*)

ABSTRACT

Previous research has shown that proper adjuvant selection is essential for maximum absorption, efficacy, and rainfastness of bispyribac-sodium. However, research is limited in regard to adjuvant systems supplied from multiple manufacturers. Research was conducted to determine the effect of methylated seed oil/organosilicone (MSO/OSL) adjuvant systems and urea ammonium nitrate (UAN) supplied from multiple manufacturers on absorption, efficacy, and rainfastness of bispyribac-sodium in barnyardgrass [*Echinochloa crus-galli* (L.) Beauv.]. Results indicate that the level of radioactivity located in the initial leaf washes was highest in the water wash. Application of ^{14}C-bispyribac-sodium with no adjuvant or nitrogen source resulted in the highest levels of radioactivity being located in the water wash, regardless of adjuvant system. The addition of Rivet® plus UAN or Sil-MES 100® plus UAN to ^{14}C-bispyribac-sodium resulted in the least amount of radioactivity located in the water wash. Accordingly, addition of these adjuvant systems resulted in the highest levels of absorption. Application of ^{14}C-bispyribac-sodium with no adjuvant system or nitrogen source resulted in significant reductions in the amount of radioactivity located in the remaining above ground foliage and roots. Bispyribac-sodium applied with no adjuvant system or with

UAN resulted in less control than with any adjuvant system. Addition of methylated seed oil (MSO) and/or MSO/OSL adjuvants plus UAN resulted in greater than 85% freshweight reduction of barnyardgrass. Barnyardgrass freshweight reduction due to application of bispyribac-sodium plus UAN only was unacceptable (<25%) regardless of simulated rainfall timing. Less than 25% control was observed for all rainfall timings. Bispyribac-sodium applied with MSO or Dyne-A-Pak® was rainfast by two and four hours after application, respectively. Addition of Sil-MES 100® plus UAN or Inergy plus UAN to bispyribac-sodium reduced the rainfastness interval to 30 minutes and one hour after herbicide application, respectively.

Nomenclature: bispyribac-sodium; barnyardgrass, *Echinochloa crus-galli* (L.) Beauv. ECHCG.

Abbreviations: ALS, acetolactate synthase; cm, centimeter; h, hour; L ha^{-1}, liters per hectare; m, meter; MSO, methylated seed oil; MSO/OSL, methylated seed oil/organosilicone adjuvant; OSL/NIS, non-ionic organosilicone adjuvant; UAN, urea ammonium nitrate

INTRODUCTION

Barnyardgrass is a troublesome annual weed in rice (*Oryza sativa* L.) (Smith 1968). Barnyardgrass germinates and emerges simultaneously with rice and competes with rice early in the growing season (Smith 1968). Competition for as little as two- to three-weeks can decrease rice yields (Smith 1968). Barnyardgrass densities greater than five plants m^{-2} require control action to prevent yield losses (Smith 1988).

Bispyribac-sodium is an acetolactate synthase (ALS) inhibitor used for control of grasses (especially *Echinochloa* spp.), sedges, and broad-leaved weeds in rice (Vencill 2002). Bispyribac-sodium has been shown to provide good to excellent control of barnyardgrass in rice while causing very little crop injury (Schmidt et al. 1999; Webster et al. 1999; Williams 1999). Moreover, bispyribac-sodium provides excellent control of barnyardgrass 20 to 40-cm tall, demonstrating its potential as a salvage tool (Williams 1999).

Identification of the appropriate adjuvant for a specific herbicide is important for maximum herbicidal activity (Wanamarta et al. 1989). The use of adjuvants is necessary for maximum absorption and efficacy of bispyribac-sodium into barnyardgrass (Dodds et al. 2007; Koger et al. 2007). The addition of methylated seed oil/organosilicone (MSO/OSL) and organosilicone/non-ionic (OSL/NIS) adjuvants to bispyribac-sodium increased absorption compared to bispyribac-sodium with no adjuvant system. Furthermore, the addition of UAN to MSO/OSL and OSL/NIS adjuvants increased absorption four- to five-fold (Dodds et al. 2007). Increases in absorption due to addition of UAN have also been observed (Beckett and Stoller 1991; Bruce et al. 1993; Bunting et al. 2004). Adjuvant systems of MSO/OSL plus UAN increased absorption of bispyribac-sodium in barnyardgrass compared to OSL/NIS adjuvant systems with and without UAN (Dodds et al. 2007). Additives do not appear to affect translocation, as the relative amount of material translocated is due to increased absorption (Beckett and Stoller 1991; Dodds et al. 2007). However, no previous research exists regarding absorption and translocation of bispyribac-sodium in barnyardgrass with MSO/OSL plus UAN adjuvants supplied from different manufacturers.

Adjuvants vary in their effectiveness and have the potential to reduce herbicide rates needed for effective weed control (Nalewaja et al. 1991). Adjuvant efficacy is a function of environment, specific herbicide, and target weed species (Penner 1989; Roggenbuck et al. 1988; Roggenbuck et al. 1990). The addition of a spray adjuvant is necessary for maximum bispyribac-sodium efficacy on barnyardgrass (Koger et al. 2007). The addition of UAN to OSL/NIS adjuvants decreased height and biomass compared to OSL/NIS adjuvants without UAN as well as MSO/OSL adjuvants with or without UAN (Koger et al. 2007). Biomass reduction of two- and five-leaf barnyardgrass increased with the addition of UAN to 11 g ai ha^{-1} of bispyribac-sodium; however, 23 g ai ha^{-1} with or without UAN controlled barnyardgrass 98 to 99% (Koger et al. 2007). Increased absorption due to addition of UAN to MSO/OSL and OSL/NIS adjuvants correlates to increases in efficacy compared to bispyribac-sodium applied without UAN (Koger et al. 2007). Increased herbicidal efficacy with the addition of UAN has been extensively discussed (Bruce et al. 1993; Hammes 1993).

The time between foliar herbicide application and rainfall is critical for the efficacy of many herbicides (Bryson 1987). However, an adjuvant that provides rainfastness for one species may not be as effective on other species (Reddy and Singh 1992). Roggenbuck et al. (1993) indicated that rainfastness may be a function of greatly accelerated herbicide penetration through the cuticle in the presence of adjuvants. Efficient herbicide uptake is related to rainfastness (Feng et al. 2000). Therefore, as with herbicide efficacy, identification of the appropriate adjuvant for a specific herbicide is important for maximum herbicidal activity and minimizing the time needed between foliar herbicide application and a rainfall event (Wanamarta et al. 1989). The use of adjuvants may not consistently improve rainfastness of all herbicides (Miller et al. 1998). Reddy and Singh (1992) indicated that specific adjuvants provide benefits specific to certain weed species. Adjuvants can greatly increase the rainfastness period of a given herbicide; however, as time between herbicide treatment and simulated rainfall increased, the differences between adjuvants may decrease (Sun et al. 1996).

Rainfall after bispyribac-sodium application affects efficacy on barnyardgrass (Koger et al. 2007). By label, bispyribac-sodium requires an 8 h rainfree period for maximum herbicidal activity (Anonymous 2006). However, the addition of OSL/NIS or MSO/OSL adjuvants and UAN reduces this rainfree period to 1 to 4 h after application (Koger et al. 2007).

No previous literature exists regarding absorption, efficacy, and rainfastness of bispyribac-sodium in barnyardgrass utilizing MSO/OSL plus UAN adjuvants supplied from multiple manufacturers. Thus, the objectives of this research were to determine the effect of MSO/OSL plus UAN adjuvants from multiple manufacturers on absorption, efficacy, and rainfastness of bispyribac-sodium when used at equivalent rates.

MATERIALS AND METHODS

^{14}C-bispyribac-sodium Adjuvant Study

Barnyardgrass seeds were planted approximately 2.5-cm deep in 40 cm^3 plastic conetainers[1] containing a 1:1 mixture of sandy loam soil purchased from a local vendor and peat[2]. Conetainers were sub-irrigated with tap water for the duration of the study. Plants were thinned to 1 plant per conetainer within 1.5-wk of emergence, and grown at 35/30°C day/night temperature. Natural light was supplemented with light from sodium vapor lamps[3] to provide a 16-hour photoperiod. Approximately 1-wk after thinning (14-

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[1] D40 (40 cm^3) Deepot Cell, Stuewe and Sons, Inc., 2290 SE Kiger Island Dr., Corvallis, OR 97333-9425.

[2] Nature's Richest Sphagnum Peat Moss. Michigan Peat Co., P.O. Box 980129, Houston, TX 77098.

[3] General Electric Sodium Vapor Lamps, Lucalox LU 400, General Electric Consumer and Industrial Lighting, 1975 Noble Rd., Nela Park, Cleveland, OH 44112.

cm tall plants), all plants were fertilized[4] with 168, 56, and 56 kg ha^{-1} (actual nitrogen, phosphorus, and potassium, respectively). The fertilization rate was based on soil test recommendations for rice production in Mississippi.

Approximately four days after fertilizing, plants with a fully expanded second true leaf were selected for treatment (15- to 20-cm in height at the newest formed leaf collar) in the first experiment. However, in the second experiment, barnyardgrass plants were significantly taller (20- to 25-cm in height at the newest formed collar) than in the first experiment. Approximately two to four-cm from the collar, 2.5-cm of the second true leaf of each plant was covered with adhesive backed paper[5] prior to spraying. Plants were pre-sprayed with non-radiolabelled bispyribac-sodium[6] at 22.5 g ai ha^{-1} plus the adjuvants listed in Table 3.1. A bispyribac-sodium with no adjuvant treatment was also included for comparison. All plants were pre-sprayed using a compressed air spray chamber with an XR11002E[7] flat fan nozzle at an application volume of 187 L ha^{-1} and a pressure of 276 kPa. Immediately after spraying plants, ^{14}C-bispyribac-sodium plus adjuvant was applied to the area covered during pre-spraying. The ^{14}C solution was prepared by dissolving ^{14}C-bispyribac-sodium [^{14}C-benzene labeled with 1.2 GBq $mmol^{-1}$ specific activity, 97.5% radio-chemical purity] in an aqueous solution of a commercial formulation of bispyribac-sodium[7] and selected adjuvant systems to give a final

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[4] Miracle Grow, Azalea, Camellia, and Rhododendron Water Soluble Plant Food, 30-10-10, Scotts Miracle-Gro Products, Inc., P.O. Box 606, Marysville, OH 43040.

[5] Post-It™ Brand Fax Transmittal Memo 7671, 3M Commercial Office Supply Division, 3M Center, St. Paul, MN 55144-1000.

[6] Regiment 80 WP; Valent U.S.A Corp., P.O. Box 8025, Walnut Creek, CA 94956-8025.

[7] TeeJet, PO Box 7900, Wheaton, IL 60189-7900.

concentration of 0.12 g ai L^{-1}. The radiochemical purity of the ^{14}C-bispyribac-sodium was verified using reversed-phase high performance liquid chromatography[8] and liquid scintillation counting techniques[9]. A 10-µl volume of the final ^{14}C-bispyribac-sodium and adjuvant solution containing 5.67 kBq was placed on the adaxial surface of the second fully expanded true leaf using a 10-µl pipette[10]. To ensure that the applied ^{14}C-herbicide solution remained in the treatment area, the treated leaf was held in a horizontal position using chenille stems, and lanolin barriers were placed traverse to each edge of the treated leaf zones.

Plants were harvested 24 h after treatment with ^{14}C-bispyribac-sodium. The treated portion of the second true leaf was excised, and ^{14}C-bispyribac-sodium remaining on the leaf surface was removed by washing in 10 ml deionized water for 15 s. Next, the treated portion of the leaf was washed in 10 ml chloroform[11] for 15 s in order to remove ^{14}C-bispyribac-sodium from the epicuticular wax. Plants were sectioned into the treated portion of the leaf, remaining above ground foliage, and roots. After washing, the treated portion of each leaf was placed in a glass scintillation vial[12] and

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[8] Hewlett Packard Series 1100 High Performance Liquid Chromatograph equipped with Chemstation software, an auto-injector, and photodiode array detector. 3000 Hanover St., Palo Alto, CA 94304-1185.

[9] Packard 1900 CA TriCarb Liquid Scintillation Counter, PerkinElmer Life and Analytical Sciences, 710 Bridgeport Ave., Sheldon, CT 06484.

[10] Gilson Pipetman, Gilson, Inc., 3000 Parmenter, P.O. Box 620027, Middleton, WI 53562.

[11] Chloroform, CX 1058-1 HPLC Grade, EM Science, 480 S. Democrat Rd, Gibbstown, NJ 08027.

[12] Disposable Glass Scintillation Vial; Wheaton Science Products, 1501 N. 10th St., Millville, NJ 08332-2093.

lyophilized[13]. Remaining plant fractions were placed in paper coin envelopes and lyophilized. A 1-ml aliquot was withdrawn from each rinsate and mixed with 10-ml of liquid scintillation cocktail[14] for quantification by liquid scintillation spectrometry. Plant samples were combusted[15], and the evolved $^{14}CO_2$ trapped in 10-ml liquid scintillation cocktail[16]. Radioactivity (dpm) in leaf washes and oxidations were quantified using liquid scintillation spectrometry[13] using internal calibration and automatic quench correction.

The sum of ^{14}C located in the leaf washes and each plant fraction was considered the amount of ^{14}C-recovered. The amount of radioactivity located in the plant fractions was considered absorbed and was expressed as percent of applied radioactivity. Radioactivity located in plant fractions other than the treated leaf was considered translocated and was expressed as percent of applied.

Treatments were arranged in a randomized complete block design. Six replications for each treatment were used in each study, and the study was repeated in time. Data were not pooled across experiments due to experiment by adjuvant interactions. Data were subjected to analysis of variance (ANOVA), and means from each experiment were separated using Fisher's protected least significance difference (LSD) test at the 5% level of probability.

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[13] Freeze Dryer, VisTis Research Equipment, Gardiner, NY 12525.

[14] Ultima Gold Universal LSC-Cocktail, PerkinElmer Life and Analytical Sciences, 549 Albany St., Boston, MA 02118-2513.

[15] OX-500 Biological Oxidizer, R.J. Harvey Instrument Company, 123 Patterson St., Hillsdale, NJ 07642.

[16] Carbon-14 Cocktail, R.J. Harvey Instrument Company, 123 Patterson St., Hillsdale, NJ 07642.

Bispyribac-sodium Efficacy Study

Barnyardgrass plants were grown and fertilized in the same manner as in the ^{14}C-bispyribac-sodium adjuvant study. Bispyribac-sodium was applied with all adjuvant systems as described above using a compressed air spray chamber with an XR11002E[8] flat fan nozzle at an application volume of 187 L ha^{-1} and a pressure of 276 kPa. Immediately after herbicide application plants were returned to the greenhouse. Fresh weights were collected 14 days after herbicide application by clipping above ground biomass at the soil surface. Percent fresh weight reductions from each replication were based on fresh weights of the untreated check from each replication. Treatments were arranged in a randomized complete block design with each treatment replicated four times and the experiment replicated in time. Data were pooled over experiment due to no interactions. Data were subjected to analysis of variance using the PROC MIXED procedure of the Statistical Analysis System (SAS® version 9.1; SAS® Institute Inc.; Cary, NC). The non-treated check was not included in data analysis. Percent freshweight reduction data were separated using Fisher's Protected LSD at $P = 0.05$.

Bispyribac-sodium Rainfastness Study

Barnyardgrass plants were grown and fertilized in the same manner as in the ^{14}C-bispyribac-sodium adjuvant study. Bispyribac-sodium was applied with all adjuvant systems as described above using a compressed air spray chamber with an XR11002E[8] flat fan nozzle at an application volume of 187 L ha^{-1} and a pressure of 152 kPa. A 2.5 cm rainfall event (7.5 cm water h^{-1} intensity) was applied using an indoor rainfall simulator to plants at 0.25, 0.5, 1, 2, 4, and 8 h after application (HAA) of bispyribac-sodium treatments. The rainfall simulator was set to deliver droplets at a height of 2 m above plants, and the amount of delivered rainfall was measured at plant level with rainfall gauges. Plants were returned to the greenhouse after rainfall simulation. A no rainfall timing treatment for each herbicide treatment was included.

Fresh weights were collected 14 days after herbicide application by clipping aboveground biomass at the soil surface. Percent fresh weight reductions from each replication are based on fresh weights of the untreated check from each replication. The experiment was conducted as a randomized complete block design with a factorial arrangement of treatments with factor A being adjuvant system and factor B being simulated rainfall timing after herbicide application. All treatments were replicated four times and the experiment was repeated three times. Barnyardgrass freshweight reduction data were pooled over three experiments due to no experiment by adjuvant by rainfall timing interactions. Data were subjected to analysis of variance using the PROC MIXED procedure of the Statistical Analysis System (SAS version 9.1; SAS Institute Inc.; Cary, NC). The non-treated check was not included in data analysis. Percent freshweight reduction data were separated using Fisher's Protected LSD at P = 0.05.

RESULTS AND DISCUSSION

^{14}C-bispyribac-sodium Adjuvant Study

Recovery levels ranged from 86 to 111% in the first experiment (Table 3.2) and 56 to 85% (Table 3.3) in the second experiment. Reduced recovery levels in the second experiment may be due to abnormally high levels of radioactivity located in the applied standards. The greatest amount of radioactivity located in the leaf washes was in the initial water wash (Tables 3.2 and 3.3). These results are in agreement with the findings of Beckett and Stoller (1991) and Dodds et al. (2007). Application of ^{14}C-bispyribac-sodium with no adjuvant and no nitrogen source resulted in increased radioactivity located in the water wash compared to applications that included an adjuvant system (Tables 3.2 and 3.3). Greater than 71% of applied ^{14}C-bispyribac-sodium was in the initial water wash when an adjuvant system was not used (Tables 3.2 and 3.3). The addition of UAN to ^{14}C-bispyribac-sodium resulted in significant reductions of radioactivity located in the water wash compared to ^{14}C-bispyribac-sodium with no adjuvant system (Tables 3.2 and 3.3). Application of ^{14}C-bispyribac-sodium plus WECO-07 resulted in similar amounts of radioactivity in the water wash to that of ^{14}C-bispyribac-sodium plus UAN (Tables 3.2 and 3.3). The least amount of radioactivity located in the water wash in both experiments was observed with application of ^{14}C-bispyribac-sodium and Sil-MES 100® plus UAN and Rivet® plus UAN (Tables 3.2 and 3.3). In the first experiment, similar levels of radioactivity in the water wash were obtained with ^{14}C-bispyribac-sodium and MSO as well as Phase II® (Table 3.2). In both experiments, radioactivity located in the water wash was similar with ^{14}C-bispyribac-sodium and Phase II® or Inergy® plus UAN (Tables 3.2 and 3.3). In both experiments, the amount of radioactivity located in the chloroform wash was highest with the application of ^{14}C-bispyribac-sodium and UAN (Tables 3.2 and 3.3). All other treatments resulted in similar amounts of radioactivity being located in the chloroform wash (Tables 3.2 and 3.3). Chloroform washes were clear, indicating that the ^{14}C did not come from ruptured epidermal cells, but from the cuticle (Beckett and Stoller 1991).

The addition of an adjuvant system to ^{14}C-bispyribac-sodium increased absorption compared to ^{14}C-bispyribac-sodium without an adjuvant or with the addition of UAN. Absorption of ^{14}C-bispyribac-sodium was least when applied with no adjuvant system or nitrogen source (Tables 3.2 and 3.3). The addition of UAN to ^{14}C-bispyribac-sodium increased absorption in both experiments compared to ^{14}C-bispyribac-sodium alone; however, absorption was reduced with the addition of UAN compared to all other adjuvant systems (Tables 3.2 and 3.3). In the first experiment, absorption was highest (74 to 78%) when Dyne-A-Pak®, Inergy® plus UAN, Rivet® plus UAN, and Sil-MES 100® plus UAN were added to ^{14}C-bispyribac-sodium (Table 3.2). Absorption of ^{14}C-bispyribac-sodium plus Dyne-A-Pak® in the first experiment was similar to that of Dodds et al. (2007). Similarly in the second experiment, absorption was highest (51 to 54%) when Rivet® plus UAN and Sil-MES 100® plus UAN were added to ^{14}C-bispyribac-sodium (Table 3.3). In the first experiment, absorption of ^{14}C-bispyribac-sodium was similar with the addition of MSO, Phase II®, and WECO-07 and ranged from 56 to 63% (Table 3.2). In the second experiment, ^{14}C absorption ranged from 22 to 30% with the addition of MSO, Dyne-A-Pak®, and Soysurf Xtra® (Table 3.3).

^{14}C-bispyribac-sodium located in the treated area was lowest when applied with no adjuvant system or UAN in both experiments (Tables 3.2 and 3.3). Radioactivity located in the treated area was greatest when ^{14}C-bispyribac-sodium was applied with Rivet® plus UAN or Sil-MES 100® plus UAN (Tables 3.2 and 3.3). In experiment one, Inergy® plus UAN provided similar levels of radioactivity in the treated area to that of Rivet® plus UAN or Sil-MES 100® plus UAN (Table 3.2). Radioactivity located in the treated area was similar in both experiments when ^{14}C-bispyribac-sodium was applied with MSO, Dyne-A-Pak®, and Soysurf Xtra® (Tables 3.2 and 3.3).

Radioactivity located in above ground foliage was greatest (30 to 37%) when ^{14}C-bispyribac-sodium was applied with MSO, Dyne-A-Pak®, Phase II®, and Soysurf Xtra® in experiment one (Table 3.2). However, in experiment two, radioactivity was greatest in the above ground foliage when ^{14}C-bispyribac-sodium was applied with Inergy® plus UAN (26%) and Rivet® plus UAN (26%) (Table 3.3). In both experiments, the least amount of radioactivity was located in the above ground foliage when ^{14}C-bispyribac-sodium was applied with no adjuvant or nitrogen source (Tables 3.2 and 3.3). The addition of UAN to ^{14}C-bispyribac-sodium resulted in similar levels of radioactivity in the above ground foliage to that of WECO-07 within each experiment (Tables 3.2 and 3.3).

^{14}C located in the roots was lowest (<0.3%) in both experiments when ^{14}C-bispyribac-sodium was applied with no adjuvant (Tables 3.2 and 3.3). The amount of ^{14}C located in the roots (<1.5%) was similar to that of Dodds et al. (2007). Similar to results obtained with above ground foliage, the greatest amount of radioactivity located in the roots in experiment one were observed when ^{14}C-bispyribac-sodium was applied with Dyne-A-Pak®, Soysurf Xtra®, and MSO (1.2 – 1.3%) (Table 3.2). In experiment two, the highest levels of radioactivity located in the roots were obtained when ^{14}C-bispyribac-sodium was applied with Rivet® plus UAN or Sil-MES 100® plus UAN (Table 3.3).

Relative translocation in both experiments was similar when ^{14}C-bispyribac-sodium was applied with no adjuvant, UAN, MSO, Dyne-A-Pak®, Phase II®, Soysurf Xtra®, and WECO-07 (Tables 3.2 and 3.3). However, in experiment one, translocation was less when ^{14}C-bispyribac-sodium was applied with Inergy plus UAN, Rivet® plus UAN, and Sil-MES 100® plus UAN (Table 3.2). In experiment two, the highest levels of translocation were obtained when ^{14}C-bispyribac-sodium was applied with Inergy® plus UAN (Table 3.3). As in experiment one, reduced amounts of ^{14}C were translocated when ^{14}C-bispyribac-sodium was applied with Sil-MES 100® plus UAN (Table 3.3).

Bispyribac-sodium Efficacy Study

Bispyribac-sodium applied without an adjuvant or nitrogen source provided only 8% freshweight reduction of barnyardgrass (Table 3.4). The addition of UAN to bispyribac-sodium did not decrease barnyardgrass freshweight compared to bispyribac-sodium applied without an adjuvant or nitrogen source (Table 3.4). The addition of any adjuvant system to bispyribac-sodium decreased freshweight 85 to 91% compared to bispyribac-sodium applied without an adjuvant or with UAN (Table 3.4). The addition of any adjuvant system to bispyribac-sodium resulted in at least 85% barnyardgrass freshweight reduction. In the case of barnyardgrass, adjuvant systems formulated with a nitrogen source and applied with bispyribac-sodium performed similarly to formulations that were not formulated with a nitrogen source, but had 32% UAN added (Table 3.4). Freshweight reductions observed when nitrogen was included with an adjuvant system and applied with bispyribac-sodium were similar to those observed by Koger et al. (2007).

Bispyribac-sodium Rainfastness Study

Bispyribac-sodium applied with UAN did not provide acceptable (<25%) freshweight reductions regardless of simulated rainfall application timing (Table 3.5). Barnyardgrass control was erratic when bispyribac-sodium was applied with UAN and subjected to simulated rainfall. Highest levels of freshweight reduction ranged from 19 to 23% (Table 3.5). Conversely, at some simulated rainfall application timings, barnyardgrass plants treated with bispyribac-sodium plus UAN were larger in size than the untreated checks (denoted as negative levels of control).

Bispyribac-sodium applied in conjunction with MSO was rainfast by two hours after application. Barnyardgrass freshweight reduction was 45% when simulated rainfall was applied 15 minutes after herbicide application. Maximum control of 71% was achieved when no rainfall was applied to plants treated with bispyribac-sodium plus MSO (Table 3.5). Bispyribac-sodium applied with Dyne-A-Pak® was rainfast by approximately two hours after application. Although freshweight reduction was somewhat lower when rainfall was applied eight hours after herbicide application, control was maximized when a two- to four-hour rainfastness period was implemented (Table 3.5). Koger et al. (2007) observed similar results when applying bispyribac-sodium and Dyne-Amic® plus UAN. No differences in barnyardgrass freshweight reduction were observed due to simulated rainfall timing when bispyribac-sodium was applied with Phase II®, Soysurf Xtra®, WECO-07, or Rivet® plus UAN (Table 3.5). Freshweight reduction with bispyribac-sodium and Inergy plus UAN was maximized (74%) when simulated rainfall was applied at least one hour after herbicide application (Table 3.5). However, control decreased somewhat as time elapsed between herbicide application and simulated rainfall application. Bispyribac-sodium and Sil-MES 100® plus UAN was rainfast by 30 minutes after herbicide application (Table 3.5). Freshweight reductions of 70% were observed when simulated rainfall was applied 30 minutes after herbicide application (Table 3.5). Control was maximized at 82% when simulated rainfall was delayed eight hours after herbicide application (Table 3.5).

Although differences were observed between experiments in relation to absorption and translocation, specific trends are evident. The addition of UAN alone to bispyribac-sodium does not provide acceptable absorption, freshweight reduction, or rainfastness. Further research is needed to determine similarities and differences in absorption and translocation between adjuvant systems that are formulated with a nitrogen source versus adjuvant systems that have UAN added to them. Herbicidal efficacy of bispyribac-sodium was maximized when methylated seed oil as well as adjuvant systems and UAN were applied to barnyardgrass. Several adjuvant systems provide a rainfastness intervals of one- to four-hours including MSO, Dyne-A-Pak®, Inergy® plus UAN, Rivet® plus UAN, and Sil-MES 100® plus UAN.

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Table 3.1. Manufacturer and constituents of adjuvants from multiple manufacturers.

Adjuvant	Manufacturer	Constituents
Urea Ammonium Nitrate (UAN)	Mississippi Chemical Company	32% UAN
Premium MSO®	Helena Chemical Company	Methylated Oils/Non-ionic surfactants
Dyne-A-Pak®	Helena Chemical Company	Alkanolamides/alkanoates/ trisiloxane/carbamides
Phase II®	Loveland Products, Inc	Carbamides/alcohol ethoxylates/methylated esters of fatty acids/polyether modified polysiloxane
Soysurf Xtra®	Jimmy Sanders, Inc.	Methylated Seed Oil/Organosilicone/UAN
WECO – 07	Wilbur-Ellis Company	Methylated Seed Oil/Organosilicone/UAN
Inergy®	Estes Inc.	Modified vegetable oil/polyalkyeneoxide modified dimethylpolysiloxane/non-ionic emulsifiers
Rivet®	Agriliance, LLC.	Methylated vegetable oil/polyether modified polysiloxane/alkyphenol ethoxylate
Sil-MES 100®	Drexel Chemical Company	Methylated Seed Oil/Organosilicone/alcohol ethoxylate

Table 3.2. Effect of adjuvant on absorption and translocation of ^{14}C-bispyribac-sodium in barnyardgrass in experiment one.[a]

Adjuvant	Water	Chloroform	Treated Area	Above Ground Foliage	Roots	Recovery	Absorption	Relative Translocation[b]
	----------	----------	----------	----------% of Applied----------	----------	----------	----------	----------
None[c]	101	3	3	3	0.3	111	7	56
Urea Ammonium Nitrate (UAN)	54	7	23	21	0.6	95	45	49
Methylated Seed Oil	21	5	32	31	1.0	89	63	50
Dyne-A-Pak®	11	3	36	37	1.3	89	75	51
Phase II®	26	4	30	30	0.6	90	60	51
Soysurf Xtra®	14	3	32	36	1.2	87	70	52

Table 3.2 (continued) Contd.

WECO-07	44	5	30	26	0.8	105	56	47
Inergy® + UAN	7	6	48	25	0.6	87	74	35
Rivet® + UAN	2	6	52	25	0.6	86	78	33
Sil-MES 100® + UAN	2	5	55	26	0.7	89	81	33
LSD (0.05)	11	3	7	9	0.4	5	10	11

[a] Plants were grown in the greenhouse prior to and after ^{14}C-bispyribac-sodium application. Greenhouse was maintained at 35/30°C day/night temperature with a 16-h photoperiod.

[b] The amount of ^{14}C translocated out of the treated leaf as a percentage of total ^{14}C absorption.

[c] Unless otherwise noted, adjuvants contain urea ammonium nitrate (UAN) as part of supplied formulation.

Table 3.3. Effect of adjuvant on absorption and translocation of ^{14}C-bispyribac-sodium in barnyardgrass in experiment two.[a]

Adjuvant	Water	Chloroform	Treated Area	Above Ground Foliage	Roots	Recovery	Absorption	Relative Translocation[b]
	% of Applied							
None[c]	71	4	1	1	0.1	76	2	50
Urea Ammonium Nitrate (UAN)	43	9	5	5	0.2	74	11	53
Methylated Seed Oil	42	2	12	10	0.2	67	22	44
Dyne-A-Pak®	53	1	15	15	0.5	85	30	52
Phase II®	28	2	21	16	0.5	68	38	44
Soysurf Xtra®	39	2	13	13	0.5	67	27	51

Table 3.3 (continued) Contd.

WECO-07	37	2	8	9	0.2	56	17	56
Inergy® + UAN	19	3	13	26	0.7	62	40	67
Rivet® + UAN	4	2	27	26	1.0	59	54	50
Sil-MES 100® + UAN	4	2	31	19	0.8	57	51	37
LSD (0.05)	9	3	5	5	0.3	14	8	13

[a] Plants were grown in the greenhouse prior to and after ^{14}C-bispyribac-sodium application. Greenhouse was maintained at 35/30°C day/night temperature with a 16-h photoperiod.

[b] The amount of ^{14}C translocated out of the treated leaf as a percentage of total ^{14}C absorption.

[c] Unless otherwise noted, adjuvants contain urea ammonium nitrate (UAN) as part of supplied formulation.

Table 3.4. Efficacy of bispyribac-sodium and methylated seed oil/organosilicone adjuvants from multiple manufacturers on barnyardgrass.[a]

Adjuvant	Rate	Fresh Weight Reduction
	(% v/v)	-------------------------%---------------------------
None[b]	--	8 B
Urea Ammonium Nitrate (UAN)	2	5 B
Premium MSO®	1	85 A
Dyne-A-Pak®	1	85 A
Phase II®	1	86 A
Soysurf Xtra®	1	89 A
WECO – 07	1	91 A
Inergy® + UAN	0.75 + 2	89 A
Rivet® + UAN	0.75 + 2	88 A
Sil-MES 100® + UAN	0.75 + 2	86 A

[a] Plants were grown in the greenhouse prior to and after ^{14}C-bispyribac-sodium application. Greenhouse was maintained at 35/30°C day/night temperature with a 16-h photoperiod.

[b] Unless otherwise noted, adjuvants contain urea ammonium nitrate (UAN) as part of supplied formulation.

Table 3.5. Barnyardgrass freshweight reductions using bispyribac-sodium plus a methylated seed oil/organosilicone adjuvant and urea ammonium nitrate.[a]

Adjuvant	Rainfall Timing (h)							Regression	R^2
	0.25	0.5	1	2	4	8	None		
	-----------------% Freshweight Reduction----------------								
Urea Ammonium Nitrate (UAN)[b]	19	3	23	-1	-3	-20	19	$Y = ((15.17/(1+(e^{-((Time – 1.516)/-0.0093)}))))$	0.49
Methylated Seed Oil	45	47	53	68	65	68	71	$Y = ((67.79/(1+(e^{-((Time + 0.2006)/0.789)})))$	0.93
Sil-MES 100 + UAN	62	70	75	72	67	82	72	$Y = ((2119/(1+(e^{-((Time – 152)/44.43)})))$	0.46
Rivet + UAN	56	54	73	67	66	69	64	$Y = ((68.47/(1+(e^{-((Time + 0.2165)/0.03583)})))$	0.66
Inergy + UAN	46	52	74	59	58	49	62	$Y = ((59.59/(1+(e^{-((Time – 0.03994)/0.1851)})))$	0.32
Phase II	60	62	73	72	66	67	72	$Y = ((69.32/(1+(e^{-((Time + 0.2578)/0.2908)})))$	0.62

Table 3.5 (continued) Contd.

Dyne-A-Pak	36	45	56	73	77	61	75	$Y = ((70.16/(1+(e^{-((Time - 0.2339)/0.4277)})))$	0.88
WECO-07	50	63	61	58	58	67	61	$Y = ((61.59/(1+(e^{-((Time - 0.2341)/0.01101)})))$	0.66
Soysurf Xtra	53	47	57	67	57	64	69	$Y = ((62.50/(1+(e^{-((Time + 0.7145)/0.7180)})))$	0.56
LSD (0.05)				18					

[a] Plants were grown in the greenhouse prior to and after ^{14}C-bispyribac-sodium application. Greenhouse was maintained at 35/30 C day/night temperature with a 16-h photoperiod.

[b] Unless otherwise noted, adjuvants contain urea ammonium nitrate (UAN) as part of supplied formulation.

CHAPTER IV

THE EFFECT OF NITROGEN FORM ON EFFICACY OF BISPYRIBAC-SODIUM PLUS A METHYLATED SEED OIL/ORGANOSILICONE ADJUVANT ON BARNYARDGRASS (*ECHINOCHLOA CRUS-GALLI*)

ABSTRACT

Previous research has shown that the addition of 32% urea ammonium nitrate to bispyribac-sodium plus methylated seed oil/organosilicone adjuvant systems can increase herbicidal efficacy and rainfastness on barnyardgrass. However, little literature exists regarding the effect of urea, ammonium, and nitrate, individually and in various combinations, on herbicidal efficacy. Experiments were conducted to determine if individual components of 32% urea ammonium nitrate as well as ammonium sulfate would provide control similar to that of 32% urea ammonium nitrate. Applications of bispyribac-sodium plus a methylated seed oil/organosilicone adjuvant without the addition of nitrogen provided less control of barnyardgrass compared to treatments that included nitrogen. Visual control data collected seven days after treatment indicate that ammonium chloride at 290 and 1190 g N ha^{-1}, calcium nitrate at 290 and 1190 g N ha^{-1}, urea at 610 g N ha^{-1}, ammonium chloride plus calcium nitrate at 290 and 290 g N ha^{-1}, and ammonium chloride plus calcium nitrate plus urea at 290 plus 290 plus 610 g N ha^{-1} all provide control similar to that of 32% urea ammonium nitrate (42%). Similarly, 14 days after treatment, both rates of ammonium chloride, the lower rate of calcium nitrate,

the combination of calcium nitrate plus ammonium chloride, and the combination of ammonium chloride plus calcium nitrate plus urea all provided similar control to 32% UAN (61%). Freshweight reductions collected 14 days after treatment mirrored visual control ratings. The addition of ammonium sulfate provided similar control at all rating periods to that of 32% UAN. These data indicate ammonium (chloride or sulfate) and calcium nitrate appear to be key components for increased herbicidal efficacy of bispyribac-sodium plus MSO/OSL for control of barnyardgrass.

Nomenclature: bispyribac-sodium; barnyardgrass, *Echinochloa crus-galli* (L.) Beauv. ECHCG

Abbreviations: AMS, ammonium sulfate; COC, crop oil concentrate; DAT, days after treatment; MSO/OSL, methylated seed oil/organosilicone adjuvant; NIS, non-ionic surfactant; UAN, urea ammonium nitrate

INTRODUCTION

The addition of urea ammonium nitrate (UAN) increases the activity of many herbicides. Wills et al. (1998) indicated that the addition of UAN at 2.34 L ha^{-1} increased the activity of imazethapyr on pitted morningglory (*Ipomoea lacunosa* L.), velvetleaf (*Abutilon theophrasti* Medik.), and barnyardgrass [*Echinochloa crus-galli* (L.) Beauv.] 11, 22, and 13%, respectively. Similarly, Fielding and Stoller (1990a) observed increased velvetleaf control with the addition of 28% UAN to thifensulfuron plus non-ionic surfactant (NIS) and chlorimuron plus NIS (Fielding and Stoller 1990b). Bunting et al. (2004) found that the addition of 28% UAN to NIS, crop oil concentrate (COC), and methylated seed oil (MSO) increased activity of foramsulfuron 47, 38, and 3%, respectively in giant foxtail (*Setaria faberi* Herrm.) and 15, 3, and 0%, respectively in woolly cupgrass (*Eriochloa villosa* (Thunb.) Kunth]. The addition of UAN to petroleum oil adjuvants increased activity of nicosulfuron 13% and primisulfuron 21% on quackgrass [*Elytrigia repens* (L.) Desv. ex B.D. Jackson] in greenhouse studies; however, these results were not obtained in field studies (Bruce et al. 1993). The addition of UAN to isoxaflutole increased giant foxtail growth reduction when applied with NIS but not with COC or MSO in greenhouse studies (Young and Hart 1998). Thifensulfuron phytotoxicity on velvetleaf was enhanced by the addition of 28% UAN (Beckett and Stoller 1991). The addition of UAN to bispyribac-sodium plus an organosilicone/non-ionic surfactant (OSL/NIS) increased phytotoxicity on barnyardgrass. Koger et al. (2007) observed 96% barnyardgrass biomass reduction when UAN was added to 11 g ai ha^{-1} bispyribac-sodium plus an OSL/NIS adjuvant system compared to 84% when UAN was not included. Koger et al. (2007) also found that the addition of UAN to either OSL/NIS or MSO/OSL adjuvant enhanced bispyribac-sodium activity; however, herbicidal efficacy on barnyardgrass was maximized when bispyribac-sodium plus OSL/NIS plus UAN was utilized.

Ammonium nitrate increases leaf absorption of 2,4,5-T in several woody species (Brady 1970) as well as 2,4-D in common sunflower (*Helianthus annuus* L.) and snap bean (*Phaseolus vulgaris* L.) (Szabo and Buchholtz 1961). However, few studies have addressed the individual components of UAN and their effect on phytotoxicity. No data exist regarding the individual nitrogen forms that comprise UAN (i.e. urea, ammonium, and nitrate) and their effect on efficacy in combination with bispyribac-sodium and an approved adjuvant system.

The inclusion of ammonium sulfate (AMS) in herbicide spray solutions is common and increases the activity of several herbicides. Wilson and Nishimoto (1975) found that the addition of AMS enhanced picloram activity on guava (*Lagenaria siceraria* var.clavata) and dwarf bean (*Phaseolus vulgaris* var. nanus) as well as strawberry guava (*Psidium cattleianum* Sabine). Enhanced activity of sethoxydim with the addition of AMS has been observed in multiple species including wild oats (*Avena fatua* L.) (Chow et al. 1983; Smith and Vanden Born 1992) and barley (*Hordeum vulgare* L.) (Smith and Vanden Born 1992). Kent et al. (1991) observed increased pitted morningglory control with imazethapyr with the addition of AMS. Further studies have shown the addition of AMS to be beneficial to glyphosate (Turner and Loader 1975; Turner and Loader 1978; Turner and Loader 1980; Wills and McWhorter 1985). However, the effect of AMS plus bispyribac-sodium and an approved adjuvant system has not been determined.

This research was conducted to determine the effects on control of barnyardgrass of urea, ammonium, nitrate, and ammonium sulfate with bispyribac-sodium plus a methylated seed oil/organosilicone adjuvant for control.

MATERIALS AND METHODS

Barnyardgrass seeds were planted approximately 2.5-cm deep in 40 cm^3 plastic conetainers[1] containing a 1:1 mixture of sandy loam soil purchased from a local vendor and peat[2]. Conetainers were sub-irrigated with tap water for the duration of the study. Plants were thinned to 1 plant per conetainer within 1.5-wk of emergence, and grown at 35/30°C day/night temperature. Natural light was supplemented with light from sodium vapor lamps[3] to provide a 16-hour photoperiod. Approximately 1-wk after thinning (14-

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[1] D40 (40 cm^3) Deepot Cell, Stuewe and Sons, Inc., 2290 SE Kiger Island Dr., Corvallis, OR 97333-9425.

[2] Nature's Richest Sphagnum Peat Moss. Michigan Peat Co., P.O. Box 980129, Houston, TX 77098.

[3] General Electric Sodium Vapor Lamps, Lucalox LU 400, General Electric Consumer and Industrial Lighting, 1975 Noble Rd., Nela Park, Cleveland, OH 44112.

cm tall plants), all plants were fertilized[4] with 168, 56, and 56 kg ha^{-1} (actual nitrogen, phosphorus, and potassium, respectively). The fertilization rate was based on soil test recommendations for rice production in Mississippi.

Within one week of fertilization, plants with a fully expanded second true leaf were selected for treatment (15- to 20-cm in height). Plants were sprayed with bispyribac-sodium[5] at 22.5 g ai ha^{-1} plus MSO/OSL adjuvant[6] and nitrogen sources and rates listed in Table 1. Nitrogen rates were selected based on previous research showing addition of 32% UAN at 0.37 L ha^{-1} at an application volume of 140 L ha^{-1} provided increased absorption of ^{14}C-bispyribac-sodium (Dodds et al. 2007). Individual and combination rates of each nitrogen species were based upon the above application rate and 32% UAN containing 16.5% urea nitrogen, 7.75% ammoniacal nitrogen, and 7.75% nitrate nitrogen. Urea[7], calcium nitrate[8], and ammonium chloride[9] were used to achieve the desired rates of each individual nitrogen form as well as combinations of each. Rates selected represent the concentration of each form present in the 32% UAN standard. Urea, calcium nitrate, and ammonium chloride rates equivalent to the total

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[4] Miracle Grow®, Azalea, Camellia, and Rhododendron Water Soluble Plant Food, 30-10-10, Scotts Miracle-Gro Products, Inc., P.O. Box 606, Marysville, OH 43040.

[5] Regiment 80 WP®; Valent U.S.A Corp., P.O. Box 8025, Walnut Creek, CA 94956-8025.

[6] Dyne-Amic®, 99% blend of polyalkyleneoxide modified polyimethylsiloxane, nonionic emulsifiers, and methylated vegetable oils, Helena Chemical Co., Suite 300, 225 Schilling Blvd., Collierville, TN 38017.

[7] Urea, Certified ACS, Fisher Scientific, Fair Lawn, NJ 07410.

[8] Calcium nitrate tetrahydrate, Certified ACS, Fisher Scientific, Fair Lawn, NJ 07410.

[9] Ammonium chloride, Certified ACS, Fisher Scientific, Fair Lawn, NJ 07410.

nitrogen concentration based on the above application rate and volume of 32% UAN were also included to quantify the effect of total nitrogen concentration on efficacy of bispyribac-sodium. A total nitrogen rate in the same ratio of the forms present in 32% UAN were produced by utilizing the individual urea, ammonium chloride, and calcium nitrate to determine if it would produce the same results as the 32% UAN or if the counter-ions that were present in each of the forms would affect herbicidal activity. Ammonium sulfate[10] rates were based on an application rate of 2.04 Kg L^{-1}. The 32% UAN was obtained from a local vendor. Remaining fertilizers used were analytical grade and were purchased from Fischer Scientific. A bispyribac-sodium plus MSO/OSL adjuvant and no nitrogen treatment was included for comparison purposes. A bispyribac-sodium treatment with no adjuvant or nitrogen source was not included because previous research indicates that an adjuvant system is required for bispyribac-sodium activity (Koger et al. 2007). In order to eliminate the effect of pH of the spray solution on nitrogen source, the pH value of the spray solutions was adjusted to that of the bispyribac-sodium plus MSO/OSL adjuvant plus UAN solution pH (Table 4.2). Spray solution pH was adjusted as needed using 0.1 M hydrochloric acid or 0.1 or 1.0 M sodium hydroxide as needed. All plants were sprayed using a compressed air spray chamber with an XR11002E[11] flat fan nozzle at an application volume of 140 L ha^{-1} and a pressure of 240 kPa.

Immediately after herbicide application the plants were returned to the greenhouse. Visual estimates of barnyardgrass control were collected seven and 14 days after treatment (DAT) and were based on a scale of 0 to 100, where 0 = no control

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[10] Ammonium sulfate, Certified ACS, Fisher Scientific, Fair Lawn, NJ 07410.

[11] TeeJet, PO Box 7900, Wheaton, IL 60189-7900.

and 100 = complete plant death (Frans et al. 1986). Fresh weights were collected 14 DAT by clipping above ground biomass at the soil surface. Percent fresh weight reductions within each replication were based on fresh weights of the untreated check within each replication. Treatments were arranged in a randomized complete block design with each treatment replicated four times and the experiment was replicated in time. Data were pooled across experiments due to no experiment by nitrogen source interactions. Data were subjected to analysis of variance using the PROC MIXED procedure of the Statistical Analysis System (SAS® version 9.1; SAS® Institute Inc.; Cary, NC). The non-treated check was not included in data analysis. Visual barnyardgrass control ratings were arcsine square-root transformed prior to analysis of variance. However, non-transformed data are presented with statistical interpretation based on transformed data. Mean visual control and percent freshweight reduction data were separated using Fisher's Protected LSD at P = 0.05.

RESULTS AND DISCUSSION

Application of bispyribac-sodium plus a MSO/OSL adjuvant with no added nitrogen resulted in less barnyardgrass control at all rating times compared to treatments that included 32% UAN or ammonium chloride plus calcium nitrate plus urea (Table 4.3). Fresh weight reductions mirrored this trend, with 65 and 69% reduction for treatments with 32% UAN and ammonium chloride plus calcium nitrate plus urea, respectively, compared to 43% freshweight reduction for treatments that did not include any nitrogen. Similar results were obtained by Koger et al. (2007) in that 68% biomass reduction of barnyardgrass was observed 14 DAT when an MSO/OSL adjuvant plus 32% UAN was added to bispyribac-sodium. Use of ammonium chloride, calcium nitrate, and urea at use rates equivalent to the total nitrogen concentration found in 32% UAN (i.e. 1190 g N ha^{-1}) provided efficacy and fresh weight reductions equivalent to those obtained with 32% UAN. (Table 4.3). This indicates that counter-ions did not affect nitrogen activity. This also confirms that the counter-ions present in the nitrogen and not the counter-ions in the ammonium chloride, calcium nitrate, or combinations of both that are responsible for improvements in herbicidal uptake into the waxy cuticle. Ammonium chloride at 290 or 1190 g N ha^{-1} provided control similar to that of 32% UAN at seven and 14 DAT. Visual control with the addition of ammonium chloride at 290 and 1190 g N ha^{-1} at 14 DAT was 55 and 57%, respectively, compared to 61 and 62% freshweight reduction, respectively (Table 4.3). Reduced control, both visually and using freshweights, with increased rates of calcium nitrate compared to 32% UAN were observed. Barnyardgrass control with the addition of calcium nitrate at 290 and 1190 g N ha^{-1} at 14 DAT was 57 and 50%, respectively, compared to 62 and 53% freshweight reduction, respectively, at the same rating period. Visual control and freshweight reductions collected 14 DAT with the addition of 32% UAN were 61 and 65%, respectively. Generally, the addition of urea at 610 or 1190 g N ha^{-1} resulted in reduced control compared to 32% UAN (Table 4.3).

Combinations of ammonium chloride plus calcium nitrate provided control equivalent to that of 32% UAN as well as ammonium chloride plus calcium nitrate plus urea (Table 4.3). However, when ammonium chloride plus urea were added to bispyribac-sodium plus MSO/OSL, reduced barnyardgrass control at all rating periods was observed compared to 32% UAN and ammonium chloride plus calcium nitrate plus urea. Visual ratings and freshweight reductions 14 DAT with ammonium chloride plus urea were 50 and 52%, respectively. Barnyardgrass freshweight reductions with the addition of calcium nitrate plus urea were significantly lower than ammonium chloride plus calcium nitrate plus urea (58% vs. 69%). The addition of ammonium sulfate provided control similar to that of 32% UAN or ammonium chloride plus calcium nitrate plus urea. Ammonium sulfate provided 42 and 58% control at seven and 14 DAT as well as 63% freshweight reduction.

Based on these data, there are several potential combinations of fertilizer products that will provide control similar to that of 32% UAN. Ammonium chloride at 290 or 1190 g N ha^{-1}, calcium nitrate at 290 g N ha^{-1}, ammonium chloride plus calcium nitrate at 290 and 290 g N ha^{-1}, ammonium chloride plus calcium nitrate plus urea at 290, 290, and 610 g N ha^{-1}, respectively, as well as ammonium sulfate at 600 g N ha^{-1} all provided similar control visually (at both seven and 14 DAT) and similar freshweight reductions as 32% UAN at 1190 g N ha^{-1}. These data indicate that not all components of UAN are providing equal activity in relation to herbicidal efficacy. Ammonium (chloride and sulfate) and calcium nitrate appear to be key components for increased herbicidal efficacy of bispyribac-sodium plus MSO/OSL.

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Table 4.1. Nitrogen sources and rates used in combination with bispyribac-sodium plus a methylated seed oil/organosilicone adjuvant for control of barnyardgrass.

	Nitrogen Form			
Nitrogen Source	NH_4	NO_3	CH_4N_2O	Total N Rate
	(g N/ha)			
32% Urea Ammonium Nitrate	290	290	610	1190
Ammonium Chloride	290	0	0	290
Ammonium Chloride	1190	0	0	1190
Ammonium Chloride + Calcium Nitrate	290	290	0	580
Ammonium Chloride + Calcium Nitrate + Urea	290	290	610	1190
Ammonium Chloride + Urea	290	0	610	900
Ammonium Sulfate	600	0	0	600
Calcium Nitrate	0	290	0	290
Calcium Nitrate	0	1190	0	1190
Calcium Nitrate + Urea	0	290	290	580
Urea	0	0	610	610
None	0	0	0	0

Table 4.2. Bispyribac-sodium and methylated seed oil/organosilicone (MSO/OSL) adjuvant plus nitrogen source spray solution pH.[a]

Constituent	Application Rate	Nitrogen Source	Nitrogen Concentration	Original pH	Adjusted pH	Original pH	Adjusted pH
			(g N/ha)	---------- Run 1 ---------		--------- Run 2 ----------	
Bispyribac-sodium	22 g ai ha^{-1}	--	--	5.77	--	6.11	--
+ MSO/OSL	0.37 L ha^{-1}	--	--	6.01	6.65	6.05	6.64
		32% Urea Ammonium Nitrate	1190	6.64	6.64	6.68	6.68
		Urea	610	6.21	6.59	6.27	6.68
		Ammonium Chloride	290	5.50	6.62	5.55	6.67
		Calcium Nitrate	290	5.94	6.63	5.94	6.67
		Urea + Ammonium Chloride	610 + 290	5.64	6.64	5.67	6.66

Table 4.2 (continued) Cont.

Urea + Calcium Nitrate	610 + 290	6.12	6.57	6.13	6.71
Ammonium Chloride + Calcium Nitrate	289 + 289	5.49	6.58	5.53	6.66
Urea + Ammonium Chloride + Calcium Nitrate	610 + 290 + 290	5.59	6.60	5.64	6.67
Urea	1190	6.49	6.65	6.67	6.67
Ammonium Chloride	1190	5.20	6.62	5.23	6.66
Calcium Nitrate	1190	5.83	6.62	5.88	6.71
Ammonium Sulfate	600	5.54	6.63	5.57	6.67

[a] pH adjustment using 0.1 M hydrochloric acid or 0.1 or 1.0 M sodium hydroxide.

Table 4.3. Efficacy of bispyribac-sodium and a methylated seed oil/organosilicone adjuvant plus various nitrogen sources.[a]

Nitrogen Source	Rate	7 DAT		14 DAT		Fresh Weight Reduction	
	(g N ha^{-1})	---%					
32% Urea Ammonium Nitrate	1190	42	ab	61	a	65	ab
Ammonium Chloride	290	43	a	55	abcd	61	abcde
Ammonium Chloride	1190	42	a	57	ab	62	abcd
Ammonium Chloride + Calcium Nitrate	290 + 290	38	abc	59	ab	59	abcde
Ammonium Chloride + Calcium Nitrate + Urea	290 + 290 + 610	43	a	60	a	69	a
Ammonium Chloride + Urea	290 + 610	33	de	50	bcd	52	def
Ammonium Sulfate	595	42	ab	58	ab	63	abc
Calcium Nitrate	290	40	abc	57	abc	62	abcde
Calcium Nitrate	1190	40	abc	50	bcd	53	cdef
Calcium Nitrate + Urea	290 + 610	36	cd	55	abcd	58	bcde
Urea	610	38	abc	48	cd	51	ef
Urea	1190	36	cd	52	abcd	57	bcde
None	--	31	e	46	d	43	f

[a] Plants were grown in the greenhouse prior to and after ^{14}C-bispyribac-sodium application. Greenhouse was maintained at 35/30°C day/night temperature with a 16-h photoperiod.