ワタのイネ科および広葉雑草に対する4-ethyl-3-(3-fluorophenyl)-1-(3-trifluoromethylphenyl)pyrrolidin-2-one(MT-141)の除草効果

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Original Article

Herbicidal efficacy of 4-ethyl-3-(3-fluorophenyl)-1-(3-trifluoromethylphenyl)pyrrolidin-2-one (MT-141) in the control of graminaceous and broad-leaved weeds in cotton

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The herbicidal activity and properties of a diphenylpyrrolidinone, MT-141 [4-ethyl-3-(3-fluorophenyl)-1-(3-trifluoromethylphenyl)pyrrolidin-2-one], were examined. MT-141 controlled barnyardgrass (*Echinochloa crus-galli*), johnsongrass (*Sorghum halepense*), green foxtail (*Setaria viridis*), large crabgrass (*Digitaria sanguinalis*), fall panicum (*Panicum dichotomiflorum*), goosegrass (*Eleusine indica*), and broadleaf signalgrass (*Brachiaria platyphylla*) at 300 g a.i./ha when applied pre-emergence (PRE), and provided greater than 90% control of these weed species at 500 g a.i./ha when applied post-emergence (POST). MT-141 was less effective against broad-leaved plants such as velvetleaf (*Abutilon theophrasti*) and ivyleaf morningglory (*Ipomoea hederacea*), but two other broad-leaved plants, hemp sesbania (*Sesbania exaltata*) and prickly sida (*Sida spinosa*), were slightly susceptible to MT-141. MT-141 applied PRE at 500 g a.i./ha did not injure cotton. The most significant herbicidal symptom for this compound was bleaching. Residual activity of MT-141 applied PRE to barnyardgrass and johnsongrass lasted at least 5 weeks. Planting depth or soil type did not affect the herbicidal activity of MT-141 at 300 g a.i./ha. MT-141 applied PRE increased the herbicidal activity of glyphosate against hemp sesbania and morningglory without injuring glyphosate-resistant cotton. Also several surfactants increased the herbicidal efficacy of this compound on POST application. MT-141 seems to be an effective herbicidal compound for controlling graminaceous weeds when applied PRE in cotton production.

Keywords: herbicidal activity, diphenylpyrrolidinone, bleaching, cotton.

Introduction

Diphenylpyrrolidinones have been reported to cause bleaching in plants.[1–3] The mechanism of bleaching was shown to be the inhibition of phytoene desaturase (PDS),[4] by using a cell-free assay with plant-type PDS prepared from cloned *Escherichia coli* transformants.[5,6] PDS, which catalyzes the dehydrogenation of phytoene to zetacarotene, is the major target enzyme of herbicides in the carotenoid biosynthesis pathway. The structural requirements of substituents on the diphenylpyrrolidinone molecule for interaction with PDS were determined by enzyme kinetic studies.[4,7,8] The study of enzyme kinetics also revealed a non-competitive inhibition of diphenylpyrrolidinones with respect to the substrate phytoene, and competitive inhibition of the cofactor $NADP^+$, suggesting an interaction of diphenylpyrrolidinones at the cofactor-binding site of PDS.[4]

PDS inhibitors such as norflurazon, fluridone, fluorochloridone, diflufenican and flurtamone are commercial herbicides.[9] The PDS-inhibitoty activity for some of these herbicides was determined[4]; $pI_{50}=6.82$ for norflurazon, $pI_{50}=7.55$ for diflufenican and $pI_{50}=6.06$ for fluorochloridone. Herbicidal usages of these compounds are as follows[9]: Norflurazon is used pre-emergence (PRE) at 1–8 kg a.i./ha to control annual grasses and broadleaved weeds in cotton and soybeans; Fluridone and fluorochloridone are applied PRE at 0.3–0.6 and 0.5–2 kg a.i./ha, respectively, to control annual weeds in cotton; Diflufenican is used at 0.125–0.25 kg a.i./ha PRE or early post-emergence (EPOST) in cereals to control grass and broadleaved weeds; and flurtamone is applied PRE or EPOST

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at 0.25–0.5 kg a.i./ha to control annual broad-leaf weeds and some grasses in cotton and cereals.

A diphenylpyrrolidinone, 4-ethyl-3-(3-chlorophenyl)-1-(3-isopropylphenyl)pyrrolidin-2-one, was characterized as a paddy rice herbicide and found to show activity against several annual weeds but not against *Scirpus juncoides* or *Sagittaria pygmaea*.[1] The PDS-inhibitory activity of this compound in a cell-free assay was $pI_{50}=7.10$.[4] Another diphenylpyrrolidinone, MT-141 [4-ethyl-3-(3-fluorophenyl)-1-(3-trifluoromethylphenyl)pyrrolidin-2-one], was discovered through a series of structure-activity studies. The PDS-inhibitory activity of MT-141 in the cell-free assay was $pI_{50}=7.30$.[4] This value is higher than that of any of the PDS inhibitors mentioned above except for diflufenican, suggesting that MT-141 may exhibit stronger weed control activity.

In this study, the herbicidal performance of MT-141 was examined under upland conditions in a greenhouse.

Materials and Methods

1. Chemicals

MT-141 [4-ethyl-3-(3-fluorophenyl)-1-(3-trifluoromethylphenyl)pyrrolidin-2-one] was synthesized as follows: *N*-(3-trifluoromethylphenyl)-2-bromo-2-(3-fluorophenyl)acetamide (1.9 g) was added to toluene (20 ml), followed by tributyltin hydride (1.2 ml) and catalytic amount of α,α-azo-bisisobutyronitrile with stirring at 70°C. After stirring for 1 hr, hydrochloric acid (200 g/l; 60 ml) was added to the reaction mixture. This mixture was then extracted with toluene. The extract was dried over anhydrous sodium sulfate, concentrated *in vacuo*, and then subjected to chromatography on a silica gel column. The 3,4-*trans*-isomer (0.9 g) and 3,4-*cis*-isomer (0.27 g) of MT-141 were obtained. The 3,4-*trans*-isomer was used after formulation as a 10% wettable powder as described previously.[10]

The surfactants used were Phase®, a blend of methylated esters of fatty acids and organosilicone fluid (Loveland Industries Inc., Greeley, CO, USA), Flame®, a blend of polyoxyethylene amine surfactant, alkyl polyoxyethylene ethers, and ethoxylated derivatives (Loveland Industries Inc.); Induce®, a blend of alkyl aryl polyoxyalkene ether, free fatty acids and dimethyl polysiloxane (Helena Chemical Company, Collierville, TN, USA); and Breakthru®, a polyether-polymethylsiloxane-copolymer (Goldschmidt Chemical Corp., Hopewell, VA, USA).

2. Evaluation of herbicidal activity

Seeds of barnyardgrass (*Echinochloa crus-galli*), johnsongrass (*Sorghum halepense*), green foxtail (*Setaria viridis*), large crabgrass (*Digitaria sanguinalis*), fall panicum (*Panicum dichotomiflorum*), goosegrass (*Eleusine indica*), broadleaf signalgrass (*Brachiaria platyphylla*), velvetleaf (*Abutilon theophrasti*), ivyleaf morningglory (*Ipomoea hederacea*), hemp sesbania (*Sesbania exaltata*), prickly sida (*Sida spinosa*) and cotton (*Gossypium hirsutum*, glyphosate-resistant cultivar, DP 436RR) were planted 0.5 cm deep in 10 cm diameter pots containing a 4 : 1 (w : w) mixture of soil [Dundee silt loam (fine-silty, mixed thermic Aeric ochraqualf) soil with pH 6.8, 1.2% organic matter, a CEC of 15.5 meq/100 g, and soil textural fractions of 25% sand, 56% silt, and 19% clay] and Jiffy Mix® (Jiffy Products of America, Batavia, IL, USA). The number of seeds planted was 50/pot for the graminaceous weeds, 20/pot for the broad-leaved weeds and 5/pot for the cotton. Pots were placed in a greenhouse at 30±2°C with a photoperiod of 14 hr. Pots were subirrigated as needed and fertilized biweekly with 50 ml of 1% (w : v) General Purpose Water Soluble Fertilizer (Scotts-Sierra Horticultural Products Corp., Marysville, OH, USA). For applications PRE, MT-141 wettable powder (WP) suspended in water was sprayed on the soil surface one day after sowing, and incorporated with artificial rain. For applications post-emergence (POST), MT-141 was applied at the 2 to 4-leaf stage of the weeds and cotton. The number of plants at the POST application was 20/pot for the graminaceous weeds, 10/pot for the broad-leaved weeds and 5/pot for the cotton.

MT-141 WP was applied with a pneumatic track sprayer (Allen Machine Works, Midland, MI, USA) with Teejet 8002 flat fan spray tips delivering 187 l/ha water at 179 kPa. MT-141 was applied at 30, 100, 300 and 500 g a.i./ha. Shoot fresh weight of each plant was measured at 3 weeks after the application (WAA).

The residual activity when MT-141 was applied PRE was also examined by measuring shoot fresh weight 1, 2, 3, 4 and 5 WAA. To examine the effect of emergence depth on the herbicidal activity, seeds were sown at a depth of 0.5, 1, 2 and 4 cm. Effect of soil type on the activity was also examined by using 2 types of soil: sandy loam (Azuma-machi, Ibaraki, Japan: total carbon, 0.4%; total nitrogen, 0.06%; pH 5.0; CEC, 8.7 meq/100 g; coarse sand, 43.0%; fine sand, 36.3%; silt, 11.8%; clay, 8.9%) and clay loam (Chonan-machi, Chiba, Japan: total carbon, 1.0%; total nitrogen, 0.11%; pH 6.0; CEC, 19.8 meq/100 g; coarse sand, 6.2%; fine sand, 40.3%; silt, 37.2%; clay, 16.3%). For the sequential application of MT-141 and glyphosate (Roundup Ultra), the former was applied one day after sowing at 300 g a.i./ha and the latter at the 2 to 4-leaf stage of the weeds and the crop at 500 and 1000 g a.i./ha. Shoot fresh weight of each plant was measured 3 weeks after the glyphosate was applied. These experiments were conducted with 2 to 3 replications and repeated.

Results and Discussion

1. Application timing and herbicidal spectrum

The herbicidal activity of MT-141 against graminaceous and broad-leaved weeds, which are important pests in cotton production, as well as the phytotoxicity of this compound to the cotton itself, was examined (Table 1). Most graminaceous weeds tested here were completely controlled by MT-141 applied PRE at 300 g a.i./ha, although johnsongrass required 500 g a.i./ha for adequate control. Among these plants, john-

Table 1. Herbicidal efficacy on selected weed species and cotton of MT-141 applied pre- and post emergence[a)]

Test plants	MT-141 (g a.i./ha)							
	Pre-emergence[b)]				Post-emergence[c)]			
	30	100	300	500	30	100	300	500
Gramineous plants								
Barnyardgrass	65±12	5±2	0	0	71±28	20±12	25±7	5±3
Johnsongrass	89±22	23±9	2±1	0	93±39	26±10	10±2	4±3
Green foxtail	14±5	4±2	0	0	44±7	19±3	7±4	6±4
Large crabgrass	22±7	9±5	0	0	55±24	15±6	6±3	6±5
Fall panicum	19±10	8±3	0	0	62±15	12±7	10±4	7±6
Goosegrass	15±12	11±6	0	0	59±9	10±7	9±3	2±1
Broadleaf signalgrass	70±26	9±3	0	0	67±19	19±2	9±5	2±1
Broad-leaved plants								
Velvetleaf	96±20	77±25	23±6	17±7	85±25	59±24	55±18	47±15
Hemp sesbania	95±16	89±18	56±15	44±31	71±11	9±4	6±1	0
Morningglory	99±36	92±27	84±29	79±21	89±40	25±12	14±2	11±6
Prickly sida	87±29	68±31	5±3	15±5	81±28	13±2	5±3	5±4
Cotton	108±28	93±17	96±21	90±14	90±16	65±23	59±17	54±14

[a)] Values are % of control±standard error based upon the fresh weight of the sample (n=4).
[b)] Fresh weight of the plants (g/pot) in untreated control; barnyardgrass (8), johnsongrass (12), green foxtail (5), large crabgrass (6), fall panicum (5), goosegrass (1), broadleaf signalgrass (2), velvetleaf (11), hemp sesbania (9), ivyleaf morningglory (10), prickly sida (6), cotton (15).
[c)] Fresh weight of the plants (g/pot) in untreaed control; barnyardgrass (14), johnsongrass (17), green foxtail (7), large crabgrass (8), fall panicum (6), goosegrass (2), broadleaf signalgrass (3), velvetleaf (19), hemp sesbania (14), ivyleaf morningglory (17), prickly sida (8), cotton (21).

songrass was less susceptible than other plant species; The remaining biomass of johnsongrass biomass was 23% at 100 g a.i./ha whereas the remaining biomass of other weeds was 11% or less at this dosage on a fresh weight basis. The results indicated that, at 30 g a.i./ha, green foxtail, large crabgrass, fall panicum and goosegrass were more susceptible to MT-141 than barnyardgrass, johnsongrass and broadleaf signalgrass, since the fresh weight of these plants was 22% or less for the former group compared with 65% or greater for the latter.

These graminaceous weeds were less susceptible to MT-141 when it was applied POST rather than PRE. They were not completely controlled with MT-141 applied POST at 500 g a.i./ha, the highest dosage used in this study. Barnyardgrass and johnsongrass were more tolerant than the other weeds, with more than 70% fresh weight remaining at 30 g a.i./ha.

MT-141 was less active against broad-leaved than graminaceous weeds on both PRE and POST application. MT-141 applied PRE had almost no herbicidal effect on velvetleaf, hemp sesbania, ivyleaf morningglory and prickly sida at 30 g a.i./ha, and was only slightly active at 100 g a.i./ha. The biomass of the weeds remaining after MT-141 was applied PRE at 500 g a.i./ha was 17, 44, 79, and 15% of that of controls, respectively. Hemp sesbania, ivyleaf morningglory and prickly sida were more susceptible to MT-141 when it was applied POST rather than PRE. In these plants, the portion of biomass remaining when MT-141 was used POST at 500 g a.i./ha was 11% or less on a fresh weight basis. In particular, hemp sesbania was completely killed at this dosage. Velvetleaf, however, was less susceptible to MT-141 on POST than PRE application; 47% of the biomass remained after MT-141 was applied POST at 500 g a.i./ha compared to 17% when MT-141 was applied PRE at the same dosage. The reason for the difference in the herbicidal activity between the PRE and POST applications in these broad-leaved weeds is unknown. MT-141 had no herbicidal effect on purple nutsedge (*Cyperus rotundus*) when used either PRE or POST at 500 g a.i./ha (data not shown).

Bleaching was the major symptom of phytotoxicity in each weed species treated with MT-141. Plants emerged with bleached leaves or cotyledons following the PRE application of MT-141, and the bleaching was observed on the treated and newly formed leaves after the POST application. After surviving with bleached leaves for several days, the weeds eventually died when sufficient dosages of MT-141 had been applied. However, at lower dosages of MT-141, treated leaves were bleached, but newly developed (formed) leaves were not bleached and plants grew normally.

Crops, including corn, rice wheat, and soybean, had unacceptable damage following treatment with MT-141. No injury was observed in cotton when MT-141 was applied PRE at 30–500 g a.i./ha (No significant difference was observed among these dosages according to an analysis of least significance difference, data not shown). However, bleaching and a subsequent reduction in growth were observed at 100–500 g a.i./ha following the POST application of MT-141.

From these results, MT-141 exhibits good herbicidal efficacy to control graminaceous weeds when applied PRE at 300 g a.i./ha without harming cotton. Other commercial PDS inhibitors had properties similar to those of MT-141. For example, norflurazon, fluridone and fluorochloridone are used PRE in cotton production.[9] These compounds are more active when applied PRE rather than POST as was the case for MT-141.

MT-141 does not seem to be translocated easily in plants, particularly to newly developed leaves (data not shown), which may account for the lower level of activity when MT-141 was applied POST in graminaceous weeds. However, the addition of surfactants to the spray increased the herbicidal activity of MT-141 applied POST as described later, indicating that MT-141 may be formulated as a commercial product for POST application.

2. *Residual activity*

The residual activity of MT-141 following its application PRE is very important for weed control because it is less active on POST application. Barnyardgrass and johnsongrass were controlled for 5 weeks after MT-141 was applied PRE at 300 g a.i./ha without any damage to cotton (Fig. 1). During this period, newly emerged plants were bleached and eventually died. On the other hand, velvetleaf was partially bleached and had reduced growth initially, but the growth tended to recover. Velvetleaf was not completely controlled during this test, because MT-141 applied PRE was less active against this weed than against barnyardgrass and johnsongrass. In a field experiment conducted at in USDA-ARS (Stoneville, MS, USA), results on the herbicidal properties of MT-141 were similar to those obtained in the greenhouse (data not shown); MT-141 showed residual activity for more than 30 days at 300 g a.i./ha, which was similar to norflurazon at 2000 g a.i./ha (data not shown).

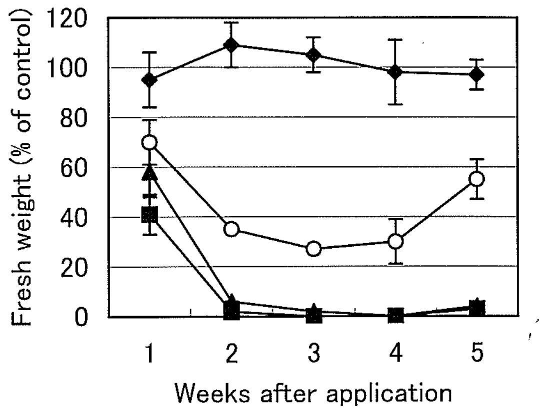

Fig. 1. Residual activity of MT-141 applied pre-emergence to barnyardgrass (■), johnsongrass (▲), velvetleaf (○) and cotton (◆). The dosage of MT-141 was 500 g a.i./ha for velvetleaf and 300 g a.i./ha for the other weeds. Vertical bars represent ±S.E.

3. *Effect of planting depth*

Barnyardgrass and johnsongrass were completely controlled by MT-141 at 300 g a.i./ha regardless of planting depth between 0.5 and 4 cm (Table 2). The activity was somewhat inconsistent at 100 g a.i./ha depending on planting depth; the herbicidal activity tended to decrease as the planting depth increased in both weed species. The decrease in activity was greater for johnsongrass. The decrease in herbicidal activity as affected by planting depth was greater in velvetleaf than in barnyardgrass and johnsongrass. The remaining biomass was 5% for velvetleaf emerging from a depth of 0.5 cm, and 49% for that emerging from 4 cm. If a herbicide is mainly absorbed

Table 2. Effect of planting depth of plants on herbicidal efficacy of MT-141 applied pre-emergence

Test plants	MT-141 (g a.i./ha)	Planting depth (cm)			
		0.5	1	2	4
Barnyardgrass	100	0	0	2±1	3±1
	300	0	0	0	0
Johnsongrass	100	10±4	19±8	15±5	26±12
	300	0	0	0	0
Velvetleaf	500	5±3	12±6	19±8	49±15
Cotton	300	104±18	101±17	90±14	105±8

Values are % of control±standard error based upon the fresh weight of the sample (n=4).

Table 3. Effect of soil type on herbicidal efficacy of MT-141 applied pre-emergence

Test plants	MT-141 (g a.i./ha)	Soil type	
		Sandy loam	Clay loam
Barnyardgrass	100	0	0
	300	0	0
Johnsongrass	100	9±2	18±7
	300	0	0
Velvetleaf	500	10±4	26±7

Values are % of control±standard error based upon the fresh weight of the sample (n=6).

through roots, then a chemical without sufficient mobility in soil to reach the roots would not be active.[11] In our preliminary tests, MT-141 was absorbed mainly from shoots and partly from roots in barnyardgrass (data not shown). The decreased activity of MT-141 against velvetleaf in response to planting depth may be due in part to differences in root forms (tap *versus* fibrous), rate of uptake by shoots, or perhaps metabolism.

4. *Effect of soil type*

Barnyardgrass was completely controlled by MT-141 applied PRE at 100 and 300 g a.i./ha in both sandy loam and clay loam, indicating little effect of soil type on the herbicidal activity against this weed species (Table 3). The effect of MT-141 on johnsongrass and velvetleaf, however, differed between the soil types, the level of activity being lower in clay loam than in sandy loam. Herbicides and allelochemicals are adsorbed on soil solids, and metabolized by chemical and biological reactions during movement in soil. This behavior is affected by various factors, such as soil texture, organic and inorganic matter, moisture and organisms, which influence the phytotoxic activity in soil.[12,13] The activity of herbicides applied to soil is also dependent upon their solubility and concentration in soil water available to the plant, which are determined by their adsorption to and desorption from the soil, and the amount of soil organic matter.[14,15] The clay loam used in this study had a higher carbon content and finer soil texture than the sandy loam. MT-141 was probably adsorbed more to components in the clay loam soil (total carbon content, 1.0%), and its concentration in soil water might be lower, resulting in less herbicidal activity than in the sandy loam (total carbon content, 0.4%). The similar organic matter-dependent (OM-dependent) change in the herbicidal activity has been reported for the case of norflurazon. The herbicidal activity of norflurazon in loamy sand containing 0.79% OM was 24 times that in silt loam containing 1.41% OM, suggesting that an increased amount of OM can reduce the herbicidal activity of norflurazon.[16]

5. *Sequential application followed by glyphosate*

Glyphosate, with a broad herbicidal spectrum, is one of the major herbicides used in cotton production in the US. Because glyphosate provides no PRE activity, the timing of its application is a critical factor in weed control. PRE herbicides reduce detrimental early season weed interference and improve flexibility for the timing of the POST application of glyphosate.[17]

Some weed species are relatively less susceptible to glyphosate. Hemp sesbania and morningglory were less susceptible to glyphosate than other weeds in our preliminary experiment. MT-141 has a potent effect on graminaceous weeds and moderate effect on broad-leaved weeds without harming cotton as shown in this study. Therefore the herbicidal effect of sequential applications of MT-141 (PRE) and glyphosate (POST) on hemp sesbania and morningglory was examined (Table 4). A single POST application of glyphosate at 500 and 1000 g a.i./ha did not completely control hemp sesbania and morningglory. Although MT-141 had insufficient activity to control these weeds following a single application, sequential applications of MT-141 and glyphosate resulted in complete control. Just before the glyphosate was applied, about 60% of the biomass of the two weeds remained when MT-141 was used PRE (data not shown). Glyphosate completely removed the remaining biomass, indicating that the sequential application of MT-141 and glyphosate was effective.

No damage to cotton was evident following these sequential applications. From these results, MT-141 was revealed to improve the control by glyphosate of hemp sesbania and morningglory.

6. *Effect of surfactant in POST application*

MT-141 applied POST did not achieve sufficient weed control (Table 1). However, the addition of a surfactant to spray solutions may improve effectiveness of MT-141. Breakthru®, Induce®, Phase® and Flame® were selected because these sur-

Table 4. Herbicidal efficacy of sequential application of MT-141 (PRE) and glyphosate (POST)

MT-141 (g a.i./ha)	Glyphosate (g a.i./ha)	Test plants		
		Hemp sesbania	Ivyleaf morningglory	Cotton
0	0	100	100	100
0	500	84±14	60±15	98±16
0	1000	79±9	6±15	93±10
300	0	31±9	56±21	90±11
300	500	0	0	89±8
300	1000	0	0	85±19

Values are % of control±standard error based upon the fresh weight of the sample (n=4).

Table 5. Effect of a surfactant in the spray solution on the herbicidal efficacy of MT-141[a)]

Test plants	MT-141 (g a.i./ha)	Surfactant (concentration in spray solution)				
		None	Breakthru (0.1%)	Induce (0.25%)	Phase (0.5%)	Flame (0.25%)
Barnyardgrass	100	26±8	21±4	30±9	4±1	28±6
Velvetleaf	100	40±14	6±5	36±6	8±4	41±7
Hemp sesbania	100	42±19	7±3	51±11	9±5	11±6
Ivyleaf morningglory	100	71±29	35±17	43±7	39±10	37±4
Prickly sida	100	5±1	0	N.T.[b)]	N.T.[b)]	N.T.[b)]

[a)] Values are % of control±standard error based upon the fresh weight of the sample (n=4).
[b)] Not tested.

factants can increase the efficacy of a herbicide depending on the weed species.[18)] Phase® was most effective in improving the herbicidal activity of MT-141 against barnyardgrass; Phase® and Breakthru® were equally effective in improving the control of velvetleaf; Phase®, Breakthru® and Flame® were equally effective in improving the control of hemp sesbania (Table 5). On the other hand, Induce® had no substantial effect on the control of any weed tested. The efficacy of MT-141 against morningglory was increased by all of the surfactants tested. These results indicate that surfactants could improve the herbicidal efficacy of MT-141 applied POST. However, the effect of these surfactants on the phytotoxicity of MT-141 in cotton needs to be clarified.

In conclusion, MT-141, a PDS-inhibiting diphenylpyrrolidinone, seems to be an effective herbicidal compound for controlling graminaceous weeds when applied PRE in cotton production.

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