Weed Science

www.cambridge.org/wsc

Herbicide residue detection in cotton as influenced by time, drift rate, and sampling method

Hannah E. Wright-Smith[1], A. Stanley Culpepper[2], Carrie R. Crabtree[3], Timothy L. Grey[2], Taylor M. Randell-Singleton[4] and Jenna C. Vance[5]

[1]Graduate Assistant, Department of Crop and Soil Sciences, University of Georgia, Tifton, GA, USA; current: Assistant Professor, Department of Horticulture, University of Arkansas, Little Rock, AR, USA; [2]Professor, Department of Crop and Soil Sciences, University of Georgia, Tifton, GA, USA; [3]Laboratory Division Director, Georgia Department of Agriculture, Tifton, GA, USA; [4]Graduate Assistant, Department of Crop and Soil Sciences, University of Georgia, Tifton, GA, USA; current: Assistant Professor, Department of Crop and Soil Sciences, University of Georgia, Tifton, GA, USA and [5]Program Technician, Department of Crop and Soil Sciences, University of Georgia, Tifton, GA, USA

Research Article

Cite this article: Wright-Smith HE, Culpepper AS, Crabtree CR, Grey TL, Randell-Singleton TM, Vance JC (2024) Herbicide residue detection in cotton as influenced by time, drift rate, and sampling method. Weed Sci. **72**: 798–803. doi: 10.1017/wsc.2024.56

Received: 27 February 2024
Revised: 1 July 2024
Accepted: 14 August 2024
First published online: 29 October 2024

Associate Editor:
Te-Ming Paul Tseng, Mississippi State University

Keywords:
Herbicide drift; pesticide residue analysis; physical drift

Corresponding author:
Hannah E Wright-Smith;
Email: hewright@uada.edu

Abstract

Herbicide drift to sensitive crops can result in significant injury, yield loss, and even crop destruction. When pesticide drift is reported to the Georgia Department of Agriculture (GDA), tissue samples are collected and analyzed for residues. Seven field studies were conducted in 2020 and 2021 in cooperation with the GDA to evaluate the effect of (1) time interval between simulated drift event and sampling, (2) low-dose herbicide rates, and (3) the sample collection methods on detecting herbicide residues in cotton (*Gossypium hirsutum* L.) foliage. Simulated drift rates of 2,4-D, dicamba, and imazapyr were applied to non-tolerant cotton in the 8- to 9-leaf stage with plant samples collected at 7 or 21 d after treatment (DAT). During collection, plant sampling consisted of removing entire plants or removing new growth occurring after the 7-leaf stage. Visual cotton injury from 2,4-D reached 43% to 75% at 0.001 and 0.004 kg ae ha^{-1}, respectively; for dicamba, it was 9% to 41% at 0.003 or 0.014 kg ae ha^{-1}, respectively; and for imazapyr, it was 1% to 74% with 0.004 and 0.03 kg ae ha^{-1} rates, respectively. Yield loss was observed with both rates of 2,4-D (11% to 51%) and with the high rate of imazapyr (52%); dicamba did not influence yield. Herbicide residues were detected in 88%, 88%, and 69% of samples collected from plants treated with 2,4-D, dicamba, and imazapyr, respectively, at 7 DAT compared with 25%, 16%, and 22% when samples were collected at 21 DAT, highlighting the importance of sampling quickly after a drift event. Although the interval between drift event and sampling, drift rate, and sampling method can all influence residue detection for 2,4-D, dicamba, and imazapyr, the factor with the greatest influence is the amount of time between drift and sample collection.

Introduction

Georgia agriculture is diverse, with many crops grown in proximity to one another. The latest farm gate value report notes that cotton (*Gossypium hirsutum* L.), fruits, peanut (*Arachis hypogaea* L.), and vegetables account for US$723 million, US$451 million, US$678 million, and US$1.2 billion, respectively (Anonymous 2022). Georgia is also unique in that there are 4.5 million ha of pine trees (*Pinus* spp.) grown for timber often located adjacent to agricultural production fields (Brandeis et al. 2016). The diversity of agriculture on Georgia farms is a fundamental component of long-term financial success. However, with such diversity, there is ample opportunity for off-target herbicide movement to damage nearby crops.

The University of Georgia (UGA) Cooperative Extension Service documented 289 agricultural drift complaints during 2014 (Culpepper et al. 2022). To foster improved pesticide stewardship decisions across Georgia agriculture, the UGA collaborated with the Georgia Department of Agriculture (GDA) to create an educational program titled "Using Pesticides Wisely" during the fall of 2014. The goal of the program was to share science-based information with growers and pesticide applicators to help them better understand how factors such as spray droplet size, sprayer speed, boom height, pesticide formulation, land terrain, wind, herbicide volatility, and so on influence pesticide movement. Additional research defining the sensitivity of commonly grown crops to various herbicides was also presented, and the importance of managing off-target pesticide movement and how illegal pesticide residues, especially in fresh market produce, jeopardize farm security were communicated. To date, 196 in-person meetings attended by 17,130 individuals account for a reduction in pesticide drift complaints by 90% (Culpepper et al. 2022).

© The Author(s), 2024. Published by Cambridge University Press on behalf of Weed Science Society of America. This is an Open Access article, distributed under the terms of the Creative Commons Attribution licence (https://creativecommons.org/licenses/by/4.0/), which permits unrestricted re-use, distribution and reproduction, provided the original article is properly cited.

Although on-target pesticide applications have greatly improved across the state, off-target events still occur each year, almost exclusively in the form of physical drift. With the adoption of auxin technologies in herbicide-resistant crops, drift issues have become even more complex, as numerous crops are sensitive to ultra-low rates of these herbicides (Bond et al. 2006; Culpepper et al. 2018; Dittmar et al. 2016; Hand et al. 2021), and detecting them through residue analysis has proven difficult.

Several factors influence the detection of herbicide residues in plant samples exposed to low-dose herbicide rates. Andersen et al. (2004) reported dicamba and 2,4-D residues in soybean [*Glycine max* (L.) Merr.] tissue decreased significantly at each sample timing, ranging from 6 to 48 d after treatment (DAT) following low-dose applications. For example, dicamba residues from a 1/10X rate or 2,4-D residues from a 1/5X rate were detected at 12 DAT but not by 24 DAT. In addition to the time interval before sample collection, herbicide rate can also influence residue detection. Lovelace et al. (2009) reported quinclorac residues in tomatoes (*Solanum lycopersicum* L.) from low-dose applications of 0.42 g ae ha^{-1} were undetectable at 3 DAT, while a higher rate, 4.2 g ae ha^{-1}, resulted in residues that were detectable up to 10 DAT. A third factor potentially influencing residue detection in plants is sampling method. Many herbicides, including dicamba, 2,4-D, and imazapyr are translocated in the plant; thus, sampling new growth may improve residue detection over whole-plant sampling (Egan et al. 2014; Nissen et al. 1995). Additionally, both Auch and Arnold (1978) and Morton et al. (1967) suggest that for systemic herbicides, sampling only meristematic portions of the plant rather than the whole plant might result in increased herbicide residue detection; however, no research evaluating this hypothesis exists.

Clearly a multitude of factors potentially influence herbicide residue detection in plant foliage following low-dose drift events. Thus, the objective of these experiments was to determine how time, simulated drift herbicide rate, and sampling method influence the choline salt of 2,4-D, the diglycolamine salt of dicamba, and the isopropylamine salt of imazapyr residue detection in non-tolerant cotton foliage. A secondary objective was to document cotton response to each simulated drift treatment. Cotton was selected as the test crop because a majority of pesticide drift complaints in Georgia over the past decade occurred in cotton (ASC, personal observation). Herbicides evaluated were strategically selected based on the highest number of incidences in agronomic production (2,4-D and dicamba) and around forest production (imazapyr) (Shepard et al. 2004; USDA 2022, 2024).

Materials and Methods

Field studies were conducted in 2020 and 2021 at the University of Georgia Ponder Research Farm near Ty Ty, GA (31.507°N, 83.657°W). Three site-years for each herbicide were evaluated: one in 2020 and two in 2021 for a total of nine field trials. Only one herbicide per experiment was evaluated in order to mitigate potential herbicide residue contamination (Table 1). Soil was of a Tifton loamy sand with 89.6% sand, 8% silt, 2.4% clay, and 0.64% organic matter with a pH of 6.3 (fine-loamy, kaolinitic, thermic Plinthic Kandiudults). This location was selected for each study because of its proximity to the GDA laboratory, allowing for immediate sample processing across numerous plant harvest dates and studies.

A three-factor factorial arranged in a randomized complete block design with four replications evaluated (1) three rates of each herbicide, (2) two time intervals between simulated drift application and plant sampling, and (3) two plant sampling technique methods. The first factor, herbicide rate, was selected with the goal of achieving maximum injury of approximately 10% to 30% with the low rate and 50% to 80% with the high rate for each herbicide. Previous literature was available to guide rate selection for 2,4-D (Byrd et al. 2016), but no such information was available for dicamba and imazapyr when treating cotton in the 8- to 9-leaf stage of growth in Georgia; therefore, rates for all herbicides were based on results of prior research for 2,4-D. Rates of 0X, 1/1,000X, and 1/250X, where X represented the typical field rates of 1.1, 0.56, and 0.56 kg ha^{-1} for 2,4-D, dicamba, and imazapyr, respectively, were evaluated in 2020. The 2,4-D rates remained constant over all studies at 0, 0.001, and 0.004 kg ha^{-1}, but the initial study for dicamba and imazapyr during 2020 indicated that the chosen rates for each herbicide led to lower levels of visual injury than desired to meet experimental objectives; thus, those two studies were only used to identify more appropriate rates to be applied during 2021. Rates during 2021 were increased to 0.003 (1/200X) and 0.014 (1/40X) kg ha^{-1} for dicamba and 0.004 (1/150X) and 0.03 (1/20X) kg ha^{-1} for imazapyr (Table 2). Only data from dicamba and imazapyr studies in 2021 are presented due to the simulated drift rate adjustment. The second factor, interval between application and plant sample harvested, consisted of 7 or 21 DAT. Field plant sampling technique was the third factor and included two levels. The first approach was the "new growth" method, which consisted of removing all plant material above node 7, focusing heavily on new growth, and the second approach was the standard method developed by GDA, which consisted of removing the entire plant at the soil surface (CRC, personal observation).

Seven studies were conducted in a conventional tillage system with two in a conservation tillage system. For conventional systems, land was prepared using a disk harrow (International Harvester, Chicago, IL), ripped and bedded (Kelley Manufacturing, Tifton, GA), and rototilled (Maletti, Modena, Italy), leaving a smooth flat surface for planting. Conservation tillage systems included desiccating a cereal rye (*Secale cereale* L.) cover crop by applying 1.3 kg ae ha^{-1} glyphosate (Roundup PowerMax® 3, Bayer CropScience, St Louis, MO), then planting through the rye using a tandem strip-till and planter (Kelley Manufacturing). For all studies, cotton was planted using a vacuum planter (Monosem, Edwardsville, KS) dropping 2 seeds every 22 cm on a row spacing of 91 cm. Each plot was 4 rows wide, with the middle two rows treated, and 9-m long. Cotton cultivar selections and planting dates are provided in Table 1. Cotton production, including pest management, growth management, and harvest, followed UGA Cooperative Extension Service recommendations (Hand et al. 2022).

Herbicide treatments were applied to 8- to 9-leaf cotton using a CO_2-pressurized backpack sprayer calibrated to deliver 140 L ha^{-1}. Low-dose herbicide rates were prepared using a serial dilution from a 1.1 kg ha^{-1} 2,4-D, 0.56 kg ha^{-1} dicamba, and 0.56 kg ha^{-1} imazapyr solution. AIXR 11002 nozzles (TeeJet® Technologies, Springfield, IL) at 207 kPa were used to apply 2,4-D and imazapyr treatments, and TTI 110015 nozzles (TeeJet® Technologies) at 276 kPa were used to apply dicamba treatments.

Data Collection and Analysis

Visual estimates of cotton injury were collected weekly using a 0% to 100% scale, where 0% is no injury and 100% is crop death, for 4 wk after application. Cotton plant heights of 20 plants $plot^{-1}$ were also measured four times over that same period. At crop maturity,

https://doi.org/10.1017/wsc.2024.56 Published online by Cambridge University Press

Table 1. Cotton cultivar, planting date, herbicide application date, sample collection dates for 7 and 21 d after treatment (DAT) for each site-year of 2,4-D, dicamba, and imazapyr residue detection studies

Herbicide	Year	Cultivar[a]	Planting	Herbicide application	7 DAT	21 DAT
2,4-D	2020	ST 5471 GLTP	May 12	June 23	June 30	July 14
	2021	ST5471 GLTP	May 2	June 10	June 17	July 1
	2021	DP 1646 B2XF	May 17	June 23	June 30	July 14
Dicamba	2021	ST 5471 GLTP	May 2	June 10	June 17	July 1
	2021	PHY 500 W3FE	May 17	June 18	June 25	July 9
Imazapyr	2021	DP 1646 B2XF	May 17	June 23	June 30	July 14
	2021	PHY 500 W3FE	May 17	June 18	June 25	July 9

[a]ST 5471 GLTP (BASF, Research Triangle Park, NC); DP 1646 B2XF (Bayer CropScience, St Louis, MO); and PHY 500 W3FE (Corteva Agriscience, Indianapolis, IN).

Table 2. Herbicide products and rates used in cotton residue detection studies

Herbicide	Trade name	Field rate	2020 rates	2021 rates
		——kg ae ha^{-1}——		
2,4-D	Enlist One® 3.8SL Corteva Agriscience, Indianapolis, IN	1.1	0 0.001 0.004	0 0.001 0.004
Dicamba	XtendiMax® 2.9SL Bayer CropScience, St Louis, MO	0.54	0 0.0005 0.002	0 0.003 0.014
Imazapyr	Arsenal® 2SL BASF Corporation, Research Triangle Park, NC	0.56	0 0.0006 0.0022	0 0.004 0.03

cotton was harvested with a spindle picker modified for small-plot harvesting (Case IH 782 Cotton Picker, Case IH, Racine, WI), and seed cotton yield was determined.

Plant tissue samples were collected using either the "new growth" method, consisting of nodes above 7, or the "whole-plant" method developed by GDA, consisting of the whole plant cut at the soil surface. At each sample timing, 3 plants per plot were removed by hand from the soil and cut at the appropriate place on the plant for the respective collection method. The three plants collected within each plot were wrapped together in aluminum foil, labeled, and placed in a matching labeled paper bag following standard procedures of the GDA. Gloves were changed and clippers were washed with methanol between plots to eliminate contamination. Samples were immediately taken to the GDA lab, approximately 20 min from the field site, and processed.

At the GDA lab, all steps were followed to ensure individual plot integrity throughout the procedure. After all foliage was removed from the stems, it was placed into a food processor (Robo Coupe U.S.A., Ridgeland, MS; Cuisinart®, Stamford, CT; Nutribullet®, Porcomia, CA) until tissue was finely chopped and homogenized. A 5-g subsample was collected from the homogenized foliage and placed into a 50-ml centrifuge tube (Hand et al. 2021). After 10 ml of water (Optima for HPLC, Fisher Scientific, Waltham, MA) was added to the tube, 300 µl of 5 N sodium hydroxide solution was added to the tube, which was then shaken using a vortex mixer rack (Benchmark Scientific, Sayreville, NJ) for 90 s at 2,500 rpm. Tubes were shaken in the manner described three times total, with 10 min between each shaking. After shaking, 10 ml of acetonitrile was added followed by 300 µl of 5 N sulfuric acid solution and vortexed for approximately 1 min. Prepackaged extraction salts (Thermo Scientific, Waltham, MA) were then added to each tube and shaken on the vortex mixer rack as previously described. Extraction salt packets consisted of 4 g of magnesium sulfate anhydrous, 1 g of sodium chloride, 1 g of trisodium citrate dihydrate, and 0.5 g of disodium citrate sesquihydrate. Tubes were shaken for 1 min then centrifuged at 3,000 rpm for 5 min. The supernatant was removed and filtered through a 0.20-µl filter into a 15-ml centrifuge tube. Subsamples from the 15-ml tube were collected and placed into 300-µl glass screw-top vials (Thermo Scientific) for analysis.

Residue analysis for each plot was performed at the GDA lab using an ExionLC™ AC (Sciex, Framingham, MA) with a Triple Quad™ 3500 Mass Spectrometer (Sciex). Mobile phases of chromatography utilized 0.1% acetic acid in water and 0.1% acetic acid in acetonitrile with an Eclipse XDB-C18 column (Agilent Technologies, Santa Clara, CA) to separate compounds.

Injury, plant height, herbicide residues, and yield were analyzed using JMP Pro 15 (SAS Institute, Cary, NC). Data were analyzed by herbicide and combined over site-years when feasible. Replication and replication nested within site-year were considered random effects, while the main effects of herbicide rate, sample collection timing, and sample collection method were fixed effects. Only the main effect of herbicide rate was evaluated for injury, plant height, and yield. The interaction between herbicide rate, sample collection timing, and sample collection method, as well as the main effects were evaluated only for herbicide residues. Data were subjected to ANOVA, and means were separated using Tukey's honestly significant difference at $\alpha = 0.05$.

Results and Discussion

2,4-D

Visual injury in the form of epinasty was initially observed within 5 DAT; however, maximum visual injury combined over site-years was observed at 21 DAT. Injury ranged from 43% from the 0.001 kg ha^{-1} rate to 75% with the 0.004 kg ha^{-1} rate (Table 3). Although leaf malformation was severe, treatments did not influence plant heights. Similarly, Sciumbato et al. (2014) also reported low-dose rates of 2,4-D did not result in a decrease in cotton plant height. Although cotton plant height may not be influenced by 2,4-D injury, cotton yield is extremely sensitive to low-dose rates of 2,4-D (Everitt and Keeling 2009). Yields in our study when combined over three locations noted a loss of 11% with the 0.001 kg ha^{-1} rate and a loss of 51% with the 0.004 kg ha^{-1} rate (Table 3); similar results were noted previously in Georgia by Byrd et al. (2016).

Although differences among years were evident (Tables 4 and 5), likely the result of environmental factors, residue analysis results highlighted the dominant implications of time interval on detectable values. For nearly all comparisons, greater levels of detection occurred with shorter sample collection time intervals. For example,

https://doi.org/10.1017/wsc.2024.56 Published online by Cambridge University Press

Table 3. Maximum injury, plant height, and seed cotton yield of 2,4-D residue detection studies[a]

Rate	Injury		Plant height[b]	Yield[c]	
kg ae ha^{-1}	—%—		———% of nontreated———		
0	0	c	100	100	a
0.001	43	b	99	89	b
0.004	75	a	101	49	c
P-value	<0.0001		NS	<0.0001	

[a]Means are separated using Tukey's honestly significant difference at α = 0.05. Means with the same letter in the same column are not different. Data are combined over 3 site-years in 2020 and 2021.
[b]Plant height averages at 21 DAT after treatment for 0, 0.001, and 0.004 treatments were 82 cm each.
[c]Yields for 0, 0.001, and 0.004 treatments were 5,102, 4,541, and 2,500 kg ha^{-1}, respectively.

Table 4. 2,4-D residues detected in cotton samples collected in 2020

Rate	Collection time[a]	2,4-D residue[b]	
kg ae ha^{-1}	DAT	——mg L^{-1}——	
0	7	0	c
	21	0	c
0.001	7	0.02	b
	21	0	c
0.004	7	0.0581	a
	21	0	c
P-value		<0.0001	

[a]DAT, days after treatment.
[b]Means are separated using Tukey's honestly significant difference at α = 0.05. Means with the same letter in the same column are not different.

during 2020, detection only occurred at 7 DAT (Table 4), while in 2021 detection was 10 to 25 times greater in three of the four relevant comparisons with samples taken at 7 DAT compared with 21 DAT (Table 5). Herbicide rate was also influential in residue detection in 2020, with 2.9 times greater residues detected with the higher herbicide rate at 7 DAT (Table 4). In 2021, for samples collected at 7 DAT, residue levels were 2 to 2.5 times higher in samples treated with the higher rate (Table 5). However, there was no difference in 2,4-D residues by 21 DAT. Sampling method did not influence residue values in 2020. However, in 2021, there was one difference when comparing sampling methods, but only when plants treated with the high rate of 2,4-D were collected at 7 DAT; the new growth approach documented more than twice the level of residue compared with the traditional whole-plant technique.

When samples were evaluated individually during 2020, 2,4-D was detected in 14 out of the 16 (88%) samples collected at 7 DAT, but 0% of samples collected at 21 DAT, when averaged over herbicide rate and sample collection method (data not shown). In 2021, 2,4-D residues were detected in 28 out of 32 (88%) samples collected at 7 DAT but only 12 out of 32 (38%) samples collected at 21 DAT, when averaged over herbicide rate and sample collection method. These findings are similar to those reported by Sirons et al. (1982), in which tomatoes treated with 1.1 g ha^{-1} 2,4-D resulted in residues of 0.01 μg g^{-1} detected at 7 DAT but no residues detected by 14 DAT.

Practically, when state agencies collect samples to make important decisions, only one or two samples are collected. Results from this experiment indicate that the time interval between drift and sample collection is the factor having the greatest level of influence on potentially documenting a drift occurrence. Thus, time is of the essence when considering management decisions regarding herbicide drift.

Table 5. 2,4-D residues detected in cotton samples collected in 2021[a]

Rate	Sample method	Collection time[b]	Residues[c]	
kg ae ha^{-1}		DAT	—mg L^{-1}—	
0	New growth	7	0	c
		21	0	c
	Whole plant	7	0	c
		21	0	c
0.001	New growth	7	0.01	b
		21	0.001	c
	Whole plant	7	0.006	bc
		21	0.0004	c
0.004	New growth	7	0.025	a
		21	0.001	c
	Whole plant	7	0.012	b
		21	0.001	c
P-value			0.0094	

[a]Data are combined over 2 site-years in 2021.
[b]DAT, days after treatment.
[c]Means are separated using Tukey's honestly significant difference at α = 0.05. Means with the same letter in the same column are not different.

Table 6. Maximum injury, plant height, and seed cotton yield for dicamba residue detection studies[a]

Rate	Injury		Plant height[b]	Yield[c]
kg ae ha^{-1}	—%—		——% of nontreated——	
0	0	b	100	100
0.003	9	b	99	104
0.014	41	a	94	100
P-value	<0.0001		NS	NS

[a]Means are separated using Tukey's honestly significant difference at α = 0.05. Means with the same letter in the same column are not different. Data are combined over 2 site-years in 2021.
[b]Plant height averages at 14 DAT after treatment for 0, 0.003, and 0.014 treatments were 64, 63, and 61 cm, respectively.
[c]Yields for 0, 0.003, and 0.014 treatments were 6,178, 6,449, and 6,108 kg ha^{-1}, respectively.

Dicamba

Visual injury consisted of epinasty and was observed within 5 DAT following low-dose treatments of dicamba. Maximum cotton injury of 9% and 41% was observed for the 0.003 and 0.014 kg ha^{-1} rates, respectively, at 14 DAT (Table 6). Dicamba did not influence plant heights or seed cotton yield. Marple et al. (2008) and Everitt and Keeling (2009) similarly observed plant recovery following low-dose applications of dicamba at 0.014 and 0.003 kg ha^{-1} to 3- to 5-leaf and 8-node cotton. Additionally, aside from tolerant cultivars, cotton is less sensitive to dicamba than to 2,4-D (Everitt and Keeling 2009).

There was an interaction between herbicide rate, sample collection method, and sample collection timing on dicamba residues detected in cotton foliage (Table 7). Similar to results observed with 2,4-D, time between application and sampling was highly impactful. In fact, for the low rate of dicamba, detection only occurred at the 7 DAT interval, while dicamba detection from the high rate was 5 to 17 times greater when collected at 7 compared with 21 DAT, as influenced by sampling method. Similarly, herbicide rate also influenced detection with 2.3 to 5.3 times higher residue values observed with the 0.014 kg ha^{-1} rate compared with 0.003 kg ha^{-1} rate but only at the 7 DAT sampling interval. Differences in sampling method were similar to those observed with 2,4-D in 2021, with the new growth sampling approach for samples treated with the higher rate and collected at 7 DAT

https://doi.org/10.1017/wsc.2024.56 Published online by Cambridge University Press

Table 7. Dicamba residues detected in cotton samples[a]

Rate	Sample method	Collection time[b]	Residues[c]	
kg ae ha^{-1}		DAT	—mg L^{-1}—	
0	New growth	7	0	d
		21	0	d
	Whole plant	7	0	d
		21	0	d
0.003	New growth	7	0.01	c
		21	0	d
	Whole plant	7	0.009	c
		21	0	d
0.014	New growth	7	0.053	a
		21	0.003	cd
	Whole plant	7	0.021	b
		21	0.004	cd
P-value			<0.0001	

[a]Data are combined over 2 site-years in 2021.
[b]DAT, days after treatment.
[c]Means are separated using Tukey's honestly significant difference at $\alpha = 0.05$. Means with the same letter in the same column are not different.

detecting more than twice the level of residue compared with the whole-plant approach.

When samples were evaluated individually, dicamba was detected in 23 out of 32 (88%) samples collected at 7 DAT but only 5 out of 32 (16%) of samples collected at 21 DAT (data not shown). As observed in the 2,4-D studies, herbicide rate and sample collection method can influence residue detection but are not as influential in detecting dicamba residues as time between the drift event and sample collection.

Imazapyr

Visual injury observed was in the form of leaf chlorosis and malformations, shortened internodes, and stunting. Injury was first observed within 3 DAT, and maximum injury was observed at 13 DAT ranging from 74% at the highest rate to only 1% with the lowest rate (Table 8). Imazapyr at 0.004 kg ha^{-1} did not reduce plant height or seed cotton yield, while the higher rate of 0.03 kg ha^{-1} reduced plant height and yield 24% and 52%, respectively. As noted by Grichar et al. (2004), cotton can be extremely sensitive to this chemistry; their work demonstrated that a 1/16X rate of imazapic or imazapyr applied preplant resulted in 60% to 81% injury at 5 wk after planting.

As with 2,4-D in 2021 and with dicamba, there was an interaction between herbicide rate, sample collection timing, and collection method on imazapyr residues (Table 9). Similarly, time interval between application and sample collection greatly influenced detection of imazapyr residues. For the lower rate, detection only occurred at the 7 DAT interval, while at the higher rate, residue values were 9 times greater when samples were collected 7 DAT as compared with 21 DAT (Table 9). Like residue results with 2,4-D and dicamba, the new growth sampling technique was more effective than the whole-plant approach, but again, only when the high rate was applied and the sample was collected at 7 DAT; it is interesting that all three chemistries had this exact same result.

Evaluating samples individually, imazapyr was detected in 22 out of 32 samples (69%) when samples were collected at 7 DAT and in 7 out of 32 samples (22%) when samples were collected at 21 DAT (data not shown). When comparing herbicide rate, imazapyr was detected at the 0.004 kg ha^{-1} rate in 7 out of 32 samples (22%)

Table 8. Maximum injury, plant height, and seed cotton yield for imazapyr residue detection studies[a]

Rate	Injury		Plant height[b]		Yield[c]	
kg ae ha^{-1}	—%—		———% of nontreated———			
0	0	b	100	a	100	a
0.004	1	b	100	a	103	a
0.03	74	a	76	b	48	b
P-Value	<0.0001		<0.0001		<0.0001	

[a]Means are separated using Tukey's honestly significant difference at $\alpha = 0.05$. Means with the same letter in the same column are not different. Data are combined over 2 site-years in 2021.
[b]Plant height averages at 14 DAT after treatment for 0, 0.004, and 0.03 treatments were 75, 75, and 57 cm, respectively.
[c]Yields for 0, 0.004, and 0.03 treatments were 8,502, 8,766, and 4,072 kg ha^{-1}, respectively.

Table 9. Imazapyr residues detected in cotton samples[a]

Rate	Sample method	Collection time[b]	Residues[c]	
kg ae ha^{-1}		DAT	—mg L^{-1}—	
0	New growth	7	0	c
		21	0	c
	Whole plant	7	0	c
		21	0	c
0.004	New growth	7	0.003	c
		21	0	c
	Whole plant	7	0.0014	c
		21	0	c
0.03	New growth	7	0.061	a
		21	0.0067	c
	Whole plant	7	0.042	b
		21	0.0049	c
P-value			0.0011	

[a]Data are combined over 2 site-years in 2021.
[b]DAT, d after treatment.
[c]Means are separated using Tukey's honestly significant difference at $\alpha = 0.05$. Means with the same letter in the same column are not different.

and at the 0.03 kg ha^{-1} rate in 22 out of 32 samples (69%) (data not shown).

These research residue analysis results align closely with Andersen et al. (2004) and Sirons et al. (1982); minimizing the time interval between a drift event and plant sampling is paramount to ensure the best opportunity for residue detection. Additionally, as the drift rate itself directly influences the potential for residue detection, understanding the importance of a shorter interval between drift event and sampling is magnified with lower drift rates. Although the current GDA sampling technique of taking the entire plant is an effective approach, altering the approach to focus on sampling primarily new growth may aid in higher detection levels of systemic herbicides, especially when shorter intervals between the drift event and plant sampling occur.

Acknowledgments. We thank the Georgia Department of Agriculture Pesticide Laboratory under the direction of CRC for the use of their facilities and collaboration in processing and analyzing samples. We especially acknowledge Dementris Williams, Corey Brooker, Jonathan Pierce, Virginia Johnson, and Jacie Mixon for their assistance. Thank you to Tim Richards for his assistance in land preparation, plot maintenance, and harvesting.

Funding statement. This research received no specific grant from any funding agency or the commercial or not-for-profit sectors.

Competing interests. No competing interests have been declared.

References

Andersen SM, Clay SA, Wrage LJ, Matthees D (2004) Soybean foliage residues of dicamba and 2,4-D and correlation to application rates and yield. Agron J 96:750–760

Anonymous (2022) 2020 Georgia Farm Gate Value Report. University of Georgia Center for Agribusiness and Economic Development. Athens: University of Georgia. 176 p

Auch DE, Arnold WE (1978) Dicamba use and injury on soybeans (*Glycine max*) in South Dakota. Weed Sci 26:471–475

Bond JA, Griffin JL, Ellis JM, Linscombe SD, Williams BJ (2006) Corn and rice response to simulated drift on imazethapyr plus imazapyr. Weed Technol 20:113–117

Brandeis TJ, McCollum J, Hartsell A, Brandeis C, Rose AK, Oswalt SN, Vogt JT, Marcano-Vega H (2016) Georgia's Forests, 2014. Resource Bulletin SRS-209. Asheville, NC: U.S. Department of Agriculture Forest Service, Southern Research Station. 78 p

Byrd SA, Collins GD, Culpepper AS, Dodds DM, Edmisten KL, Wright DL, Morgan GD, Baumann PA, Dotray PA, Manuchehri MR, Jones A, Grey TL, Webster TM, Davis JW, Whitaker JR, *et al.* (2016) Cotton stage of growth determines sensitivity to 2,4-D. Weed Technol 30:601–610

Culpepper AS, Sosnoskie LM, Shugart J, Leifheit N, Curry M, Gray T (2018) Effects of low-dose applications of 2,4-D and dicamba on watermelon. Weed Technol 32:267–272

Culpepper AS, Vance JC, Randell TM, Wright HE (2022) Pesticide drift complaints decline while dicamba technology adoption increases. Page 105 *in* Proceedings of the Weed Science Society of America Annual Meeting. https://wssa.net/wp-content/uploads/2022-WSSA-CWSS-Proceedings-Final-April-4.pdf?

Dittmar PJ, Ferrell JA, Fernandez JV, Smith H (2016) Effect of glyphosate and dicamba drift timing and rate in bell pepper and yellow squash. Weed Technol 30:217–223

Egan JF, Barlow KM, Mortensen DA (2014) A meta-analysis on the effects of 2,4-D and dicamba drift on soybean and cotton. Weed Sci 62:193–206

Everitt JD, Keeling JW (2009) Cotton growth and yield response to simulated 2,4-D and dicamba drift. Weed Technol 23:503–506

Grichar WJ, Baughman TA, Dotray PA, Lemon RG, Senseman SA (2004) Cotton response to imazapic and imazethapyr residues following peanut. Texas J Agric Nat Resour 17:32–39

Hand C, Culpepper S, Harris G, Kemerait B, Liu Y, Perry C, Hall D, Porter W, Roberts P, Smith A, Virk S, Bag S, Sintim H (2022) Georgia Cotton Production Guide. Athens: University of Georgia Cooperative Extension Service Annual Publication 124-2. 166 p

Hand LC, Vance JC, Randell TM, Shugart J, Gray T, Luo X, Culpepper AS (2021) Effects of low-dose applications of 2,4-D and dicamba on cucumber and cantaloupe. Weed Technol 35:357–362

Lovelace ML, Hoagland RE, Talbert RE, Scherder EF (2009) Influence of simulated quinclorac drift on the accumulation and movement of herbicide in tomato (*Lycopersicon esculentum*) plants. J Agric Food Chem 57:6349–6355

Marple ME, Al-Khatib K, Peterson DE (2008) Cotton injury and yield as affected by simulated drift of 2,4-D and Dicamba. Weed Technol 22:609–614

Morton Hl, Robison ED, Meyer RE (1967) Persistence of 2,4-D, 2,4,5-T, and dicamba in rage forage grasses. Weeds 15:268–274

Nissen SJ, Masters RA, Thompson WM, Stougaard RN (1995) Absorption and fate of imazapyr in leafy spurge (*Euphorbia esula*). Pestic Sci 45:325–329

Sciumbato AS, Senseman SA, Steele GL, Chandler JM, Cothren JT, Kirk IW (2014) The effect of 2,4-D drift rates on cotton (*Gossypium hirsutum* L.) growth and yield. Plant Health Progr 15:67–73

Shepard JP, Cerighton J, Duzan H (2004) Forestry herbicides in the United States: an overview. Wildlife Soc B 32:1020–1027

Sirons GJ, Anderson GW, Frank R, Ripley BD (1982) Persistence of hormone-type herbicide residue in tissue of susceptible crop plants. Weed Sci 30:572–578

[USDA] U.S. Department of Agriculture (2022) 2021 Agricultural Chemical Use Survey: Cotton. Washington, DC: U.S. Department of Agriculture. 2 p

[USDA] U.S. Department of Agriculture (2024) 2023 Agricultural Chemical Use Survey: Soybeans. Washington, DC: U.S. Department of Agriculture. 2 p

https://doi.org/10.1017/wsc.2024.56 Published online by Cambridge University Press