

**STUDY TITLE**

Magnitude of the Residue of Disulfoton in Wheat and Soybean  
Raw Agricultural Commodities

**STUDY NUMBER**

ERS23059

**DATA REQUIREMENT**

OPPTS 860 1000 Residue Chemistry Test Guidelines  
OPPTS 860 1500 Crop Field Trials

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**FIELD REPORT COMPLETION DATE**

January 15, 2004

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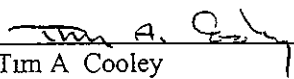
## GOOD LABORATORY PRACTICE STATEMENT

The field phase reported herein, "Magnitude of the Residue of Disulfoton in Wheat and Soybean Raw Agricultural Commodities," was conducted and reported in compliance with EPA FIFRA Good Laboratory Practice Standards (GLP) as defined in 40 CFR Part 160, except for the items indicated below

- Supporting data such as field pesticide history, in-life and historical weather data, crop maintenance, irrigation data, plot slope and soil conservation service data
- Test substance was not characterized No Certificate of Analysis provided

This did not affect the integrity of the study

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\_\_\_\_\_  
Date

## QUALITY ASSURANCE STATEMENT

Reviews conducted by the Quality Assurance Unit confirm that the field data and summary report reflects the raw data for the field phase of the study. The final field summary report was inspected by Vincella J. Erickson on January 3, 2004, and accurately reflects the study as it was conducted.

The following is a list of reviews conducted by Field Test Site Quality Assurance and Field Research Management Quality Assurance (Excel Research Services, Inc) on the field phase of the study reported herein.

Site	Phase	Inspection Date	Inspected By	Date Reported to Study Director	Date Reported to Management
ND01	Calibration/Application	07/21/03	SDK	09/15/03	09/15/03
	Field Logbook	12/01/03	VJE	01/15/04	01/15/04
ND02	Calibration/Application	07/21/03	SDK	09/15/03	09/15/03
	Field Logbook	12/04/03	VJE	01/15/04	01/15/04
IA02	Plot 2 Calibration/Application	06/13/03	PEJ	06/19/03	06/19/03
	Field Logbook	01/02/04	VJE	01/15/04	01/15/04

*Vincella J. Erickson*

Vincella J. Erickson  
Quality Assurance Officer

*15 Jan 2004*

Date

Quality Assurance Personnel

VJE = Vincella J. Erickson, contracted by Excel Research Services, Inc  
 SDK = S. Derek Killilea, Ph.D., contracted by Northern Plains Ag Research  
 PEJ = Patricia E. Johnston, Quality Assurance Services, contracted by Bennett Ag Research

## CERTIFICATION OF AUTHENTICITY

This report is an accurate and authentic representation of the conditions and results of the field phase of this study

Author Tim A. Cooley 15 Jan 04  
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## I SUMMARY

This magnitude of the residue study was conducted to provide raw agricultural commodity (RAC) samples of wheat and soybeans following applications of Disulfoton (D1-Syston® 8 and D1-Syston® 15%) Samples will be analyzed for the effect of processing on pesticide residues

The intent of this study was to follow the requirements under OPPTS Test Guidelines 860 1000 Residue Chemistry Test Guidelines and 860 1500 Crop Field Trials This study will closely follow EPA, FIFRA, Good Laboratory Practice Standards (GLP), 40 CFR, Part 160 (October 1989) Because the Sponsor is not the manufacturer of and has no control over the test substances used (for example, certificate of analysis availability), this study was done following GLP Standards in intent, but is not considered to be fully GLP compliant

This study was conducted to determine residue levels on wheat and soybean raw agricultural commodity (RAC) samples following one application of Disulfoton (D1-Syston® 8) to two different varieties of wheat at 30 days PHI at two sites, one application of Disulfoton (D1-Syston® 15% Granule) to soybeans at planting at one plot, and one application of Disulfoton (D1-Syston® 8) to soybeans at early flower stage at another plot The applications of D1-Syston® 8 to wheat were made at a target rate of 60 fl oz product/A and to soybeans at 10 pints/A The application of D1-Syston® 15% was made at a target rate of 40 oz per 1000 foot of row The actual applications rates ranged between 98.5 to 101.2 percent of target D1-Syston® 8 was applied at spray rates ranging from 15 to 29 gallons per acre (GPA)

The field phase of this study was conducted from June 13, 2003, through November 17, 2003, corresponding to the first application through the last date of sample shipment This study was conducted at three sites representing United States EPA Region V, typical wheat and soybean-growing areas

Weather conditions during the trial period were typical for the regions Rainfall was adequate for normal growth and development, therefore, no irrigation was applied Agronomic practices, including fertilizer use and maintenance practices, were typical for wheat and soybean production at each site

Wheat and soybean samples were successfully collected at all sites Wheat and soybean samples were stored frozen and shipped on dry ice via FedEx to Japan

## II INTRODUCTION

Disulfoton is formulated as a liquid (D1-Syston® 8, 8 pounds ai per gallon) and granular (D1-Syston® 15%) Disulfoton is an insecticide used to control sucking insects in cereal and other crops This study was conducted to provide raw agricultural commodity samples of wheat following one boom sprayer application of D1-Syston® 8 at 30-day pre-

harvest interval on wheat, and to soybeans following one application of D1-Syston® 15% at planting, and on a separate plot, one application of D1-Syston® 8 as a side dressing after plants became established. This study was designed to support the application for pesticide label registration under the Environmental Protection Agency's Residue Chemistry Test Guidelines, OPPTS 860 1000 and 860 1500, Crop Field Trials, in accordance with EPA FIFRA Good Laboratory Practice (GLP) Standards, 40 CFR, Part 160. This report summarizes the procedures and data generated in the field phase of the study.

### III FIELD PROCEDURES

#### A Test Substance Description

Trade Name	Disulfoton
Product Formulation	D1-Syston® 8 (liquid)
Active Ingredient (nominal)	O,O-Diethyl S-[2-(ethylthio)ethyl]-phosphorodithioate
CAS Number (a1)	298-04-4
Lot Number (ND01 & ND02)	9-01-0718
Expiration Date	Not known
Lot Number (IA01)	9-03-0186
Expiration Date	05/16/04
Appearance	Clear/amber liquid

\*\*\*\*\*

Trade Name	Disulfoton
Product Formulation	D1-Syston® 15% (granular)
Active Ingredient (nominal)	O,O-Diethyl S-[2-(ethylthio)ethyl]-phosphorodithioate
CAS Number (a1)	298-04-4
Lot Number	0-03-0127
Expiration Date	05/16/04
Appearance	Brown granules

The receipt of the test substance by each Field Principal Investigator was recorded in the field notebooks.

#### B Test System

The test system was wheat and soybean grown in a typical growing region.

## C Field Test Sites

### 1 Overview

The field phase of this study was comprised of three sites located in the major wheat and soybean-producing areas representing United States EPA Region V

Field Principal Investigators for each site were chosen for their capabilities in conducting regulatory field trials and for their ability to obtain trial sites in the wheat and soybean-growing region. The site codes, locations, and Field Principal Investigators are listed in Table 1. A map of the United States showing the test site locations is presented in Figure 1.

The layout at Sites ND01 and ND02 (wheat) consisted of a single untreated plot and a single treated plot. Site IA01 (soybean) contained a single untreated and two treated plots, one of which was treated with the granular formulation and one with the liquid formulation. Wheat and soybean samples were successfully collected at all sites.

### 2 Plot Size, Site Descriptions, Crop Growth, and Development

For each test site, field pesticide histories, including crops grown and pesticides used, were obtained for the previous year. Field pesticide history information is presented in Table 2.

The soil type at all sites was silty clay loam and was typical of the soils in the major wheat and soybean-growing areas representative of United States EPA Region V. Soil type information is presented in Table 3.

At Sites ND01 and ND02, the untreated plots were 20 feet by 225 feet and the treated plots were 40 feet by 170 feet. At both sites, the untreated plot was positioned more than 100 feet from the treated plot. At Site IA01, the untreated and treated plots were 30 feet by 100 feet. The untreated plot was positioned 50 feet from the treated plot 2 and more than 100 feet from treated plot 3. The treated and untreated plots were identified by uniquely coded flags. Plot maps are presented in Figures 2 through 4. Field test site layout information is presented in Table 4.

Test site preparation and maintenance (including pesticides used, agronomic practices employed, and fertilizers applied) were



recorded. Test site preparation and maintenance information is presented in Table 5.

Crop variety and planting information is presented in Table 6.

### 3 Test Substance Treatment Rate and Application Timings

At Sites ND01 and ND02 (wheat) the D<sub>1</sub> Syston 8 (liquid) was applied at 30 days before harvest at a target rate of 60 fl oz product/A. The actual application rates were 98.5 percent of target at ND01 and 101.2 percent of target at ND02.

At Site IA01 (soybean) plot 2, D<sub>1</sub>-Syston 15% granular was applied at planting (123 days before harvest) at a target rate of 40 oz/1000 ft row. The actual application rate at plot 2 was 100 percent of target. At Site IA01 (soybean) plot 3, D<sub>1</sub> Syston 8 (liquid), was applied at 82 days before harvest at a target rate of 10 pints/A. The actual application rate at plot 3 was 98.5 percent of target. Test substance shipping and storage information is presented in Table 7.

### 4 Application Procedures

At both sites, the application equipment was calibrated prior to application of the test substance. All applications, except the IA01 Plot 2 granular application, were verified by the time/volume technique, which is based on the output per time and equipment travel speed. The calibrations were conducted the day before or the same day as the applications at all sites. Equipment calibration and application information is presented in Table 8, and product rate determination is presented in Table 10.

The equipment used was typical of small plot research equipment that closely simulates commercial equipment. A compressed air tractor-mounted boom sprayer was used to make the applications at ND01 and ND02. A two-row JD7000 planter was used to make the application at IA01 plot 2 and a CO<sub>2</sub> row cultivator was used to make the application at plot 3. Application equipment information is presented in Table 9 and environmental conditions at application are presented in Table 11.

No problems occurred during applications.

5 Weather Data

Weather data for each site, including minimum/maximum temperatures and daily rainfall, were recorded for the trial period from permanent weather stations located near the test sites, and compared to the historical norm. Weather data are presented in Table 12.

6 Sampling and Sample Shipment

At all sites, wheat and soybean samples were successfully collected. In all cases the untreated plots were sampled before the treated plots and disposable gloves were used and changed between plots and samples.

At Sites ND01 and ND02 a Massey Harris 35 Combine with an 8-foot straight header was used for harvesting the wheat samples. The combine was cleaned thoroughly using compressed air before use. ND01 and ND02 untreated plots were sampled before ND02 and ND01 treated plots. Two passes were made through each plot, avoiding a 3-foot wide border around the plot. Samples were bagged in plastic-lined cloth residue bags and labeled with pre-printed labels before moving to the next plot. Passes were made to flush the combine between plots. Samples from ND01 and ND02 were transported from the field to the storage facility in separate coolers. Ice packs were not used because the test site was next to the storage facility. Samples were cleaned using a fanning mill before being placed in the freezer. The fanning mill was thoroughly vacuumed before use. The untreated were cleaned first, then the treated. Samples were placed in freezers and stored frozen until shipment to the Sponsor via FedEx airport to airport service. The samples at Site ND01 and ND02 weighed 35 lb each.

At Site IA01 a JD 4400 combine was used to collect the seed sample. Prior to the beginning of sample collection, the outside two rows on each side of the plot along with the plot ends were cut off to flush the combine. The control plot was sampled first followed by plot 2 and then plot 3. After the combine was flushed, half of the plots were cut with combine pass 1 and grab samples of the soybeans were collected as the grain entered the grain tank of the combine. A second bag was filled in a similar fashion with combine pass 2. Following the collection of the sample material, the combine was stopped and sample material was weighed to make sure the minimum sample material was collected. Samples were bagged in plastic-lined cloth residue bags, labeled with pre-

printed labels, and placed in coolers on “Blue Ice” for transport back to the facility freezers. Samples were transported in separate coolers, with each sample made up of two bags. Grain moisture was tested with a grain moisture meter and was 13.7% at harvest. Impartial sampling was assured by collecting grab samples as the combine made two passes through each plot.

Sampling information is presented in Table 13, and sample storage and shipping information is presented in Table 14.

## 7 Crop Destruction

At Sites ND01 and ND02 the treated plot crop was dropped down to the ground using a bushhog mower. At Site IA01 the remaining crop was harvested and extra seed material was placed on the plot area to degrade naturally.

## D **Deviations**

One deviation occurred during the field phase of this study. This deviation was documented and reported to the Study Director and had no impact on the results of this study.

### Deviation No. 1

At Site IA01, the control plot was planted 50 feet upslope from the closest treated plot. Protocol suggests a 100-foot buffer between the control and treated plots.

## E **Quality Control**

Quality control measures taken to maintain sample integrity and to avoid contamination at the field test sites were recorded in the field notebooks and include the following:

- Plots were laid out with adequate buffer zones and situated in a manner designed to minimize contamination due to drift and run-off of the test substance. The untreated plot was sampled before the treated plot at each test site.
- All samples were labeled, stored and shipped in a manner designed to prevent contamination and decomposition of the samples. Control and treated samples were boxed separately for shipment and were accompanied in transit with appropriate chain-of-custody forms.
- Access to the field plots, chemical storage, residue sample storage, trial records, etc., was restricted to authorized personnel.

**F Storage of Raw Data**

Study specific raw data generated at the field test sites will be transferred to the Sponsor upon acceptance of the final field summary report. Non study-specific raw data generated at the field test sites will be maintained with each of the field investigators.

## IV TABLES

**Table 1 Site Codes, Crop, Locations and Field Principal Investigators**

Site Code & Crop	Site Location (City, State, County, CA Region)	Field Principal Investigator
ND01 Wheat	Gardner, North Dakota Cass County EPA Region V	Quan Zai Jia, Ph D Northern Plains Ag Research 16458 19 <sup>th</sup> Street SE Gardner, ND 58036
ND02 Wheat	Gardner, North Dakota Cass County EPA Region V	Quan Zai Jia, Ph D Northern Plains Ag Research 16458 19 <sup>th</sup> Street SE Gardner, ND 58036
IA01 Soybean	Richland, Iowa Jefferson County EPA Region V	David Bennett Bennett Ag Research 1109 Ivy Avenue Richland, IA 52585



**Table 2 Field Pesticide History**

Site Code	Year	Crop	Product/ Formulation	Active Ingredient(s)	Rate (lb ai/A)
ND01	06/02	Soybean	Roundup 35L	Glyphosate-ipa salt	0.75
	07/02	Soybean	Roundup 35L	Glyphosate-ipa salt	0.50
ND02	06/02	Soybean	Roundup 35L	Glyphosate-ipa salt	0.75
	07/02	Soybean	Roundup 35L	Glyphosate-ipa salt	0.50
IA01	06/02	Soybean	Roundup Ultra Max	Glyphosate	1.0

**Table 3 Soil Type**

Site Code	Soil Series Type
ND01	Hegne-Fargo Silty Clay Loam
ND02	Hegne-Fargo Silty Clay Loam
IA01	Mahaska Silty Clay Loam

**Table 4 Field Test Site Layout**

Site Code	Control Plot Width x Length in Ft (Area in Square Feet) (Area in Acres)	Treated Plot Width x Length in Ft (Area in Square Feet) (Area in Acres)	Treated Plot Width x Length in Ft (Area in Square Feet) (Area in Acres)
	Plot 1	Plot 2	Plot 3
ND01	20 x 225 (4500) (0 1033)	40 x 170 (6800) (0 1561)	
ND02	20 x 225 (4500) (0 1033)	40 x 170 (6800) (0 1561)	
IA01	30 x 100 (3000) (0 0689)	30 x 100 (3000) (0 0689)	30 x 100 (3000) (0 0689)

**Table 5 Field Test Site Preparation and Maintenance**

Site Code	Date	Pesticide (Product/Active Ingredient)	Rate (lb ai/A)	Date	Agronomic Practice	Date	Fertilizer	Rate (lb/A)
ND01	06/16/03	Puma IEC/ Fenoxaprop-P	0.04		None	05/15/03	Urea 46% Granular 46-0-0	60
	06/16/03	Sterling 4SL/ Dicamba-dma salt	0.5			05/15/03	MAP Granular 11-56-0	20
	07/15/03	Tilt EC/ Propiconazole	0.11			05/15/03	Urea 46% Granular 46-0-0	60
ND02	06/16/03	Puma IEC/ Fenoxaprop-P	0.04		None	05/15/03	MAP Granular 11-56-0	20
	06/16/03	Sterling 4SL/ Dicamba-dma salt	0.5			05/15/03	Urea 46% Granular 46-0-0	60
	07/15/03	Tilt EC/ Propiconazole	0.11			05/15/03	MAP Granular 11-56-0	20
IA01	06/26/03	Touchdown/ Glyphosate	0.75	11/11/02	Sub soil	11/01/02	N-P-K	0-35-45
	07/31/03	Roundup Weather Max/Glyphosate	0.94	04/15/03	Field cultivate			
				05/07/03	Field cultivate			
				05/27/03	Field cultivate			
				05/28/03	Mulch			
			07/22/03	Cultivate for weeds				



**Table 6 Crop**

Site Code	Variety	Planting Date	Row Spacing (inches)	Plant Spacing (inches)
ND01	Walworth Wheat	05/23/03	6.5	0.5
ND02	Alsen Wheat	05/23/03	6.5	0.5
IA01	93B68 Soybean	06/13/03	30	1-2

**Table 7 Test Substance Receipt and Storage**

Site Code	Product/ Formulation	Amount of Test Substance	Date Test Substance Obtained <sup>1</sup>	Test Site Storage Temp <sup>2</sup>	
				Min °C	Max °C
ND01 ND02	D1-Syston® 8 (8 lb/gal EC)	1 liter	05/14/03	10.2	27.9
IA01	D1-Syston® 15% (Granule)	10 lb	05/16/03	13.67	23.67
	D1-Syston® 8 (8 lb/gal EC)	1000 mL	05/16/03	13.67	28.33

<sup>1</sup>Test substance was obtained locally by Principal Field Investigator

<sup>2</sup>Storage temperatures represent time from receipt of test substance by Principal Investigator to last application

**Table 8 Equipment Calibration and Application**

Site Code And Crop	Plot Number	Application Number	Calibration Date	Calibrated GPA	Application Date
ND01 Wheat	2	1	07/21/03	15	07/21/03
ND02 Wheat	2	1	07/21/03	15	07/21/03
IA01 Soybean	2	1	06/13/03	40 oz/ 1000 ft row	06/13/03
	3	1	07/24/03	29.7	07/24/03

**Table 9      Application**

Site Code/ Plot	App No	Equipment Type	Nozzle Type	Nozzle Tip No	Nozzle No	Nozzle Spacing (inches)	Pressure Source	Approx Pressure (psi)
ND01 Plot 2	1	Tractor-mounted Boom Sprayer	Flat Fan	80015 EVS	12	20	Compressed Air	30
ND02 Plot 2	1	Tractor-mounted Boom Sprayer	Flat Fan	80015 EVS	12	20	Compressed Air	30
IA01 Plot 2	1	2 row JD7000 planter	N/A	N/A	N/A	N/A	N/A	N/A
IA01 Plot 3	1	Row Cultivator	Micro-Tube Black	N/A	4	N/A	CO <sub>2</sub>	19

N/A=Not applicable

**Table 10 Product Rate Determination**

Site Code And Plot	Date	App No	Volume			Total Pass Time (sec)	Calibrated Spray Rate (mL/sec)	Spray Mix Applied To Plot (mL) <sup>1</sup>	Treated Area (Acres)	Spray Rate (GPA) <sup>2</sup>	Rate	
			Test Substance (mL)	Carrier (mL)	Total Mixture (mL)						Actual Rate <sup>3</sup>	% of Target <sup>4</sup>
ND01 Plot 2	07/21/03	1	407	12629	13036	93.56	93.2	8719.8	0.1561	14.8	59 fl oz product/A <sup>3</sup>	98.5
ND02 Plot 2	07/21/03	1	407	12629	13036	96.31	93.2	8976.1	0.1561	15.2	60.7 fl oz product/A <sup>3</sup>	101.2

<sup>1</sup>Spray Mix Applied to Plot (mL) = Total Pass Times (sec) x Calibrated Spray Rate (mL/sec)

<sup>2</sup>Spray Rate (GPA) =  $\frac{\text{Spray Mix Applied to Plot (mL)}}{3785 \text{ mL/gal}} \times \frac{1}{\text{Treated Area (acres)}}$

<sup>3</sup>Disulfoton =  $\frac{\text{Test Substance in Spray Mixture (mL)}}{\text{Total Mixture Volume (mL)}} \times \text{Actual Spray Rate (GPA)} \times \frac{128 \text{ fl oz}}{1 \text{ gal}}$

<sup>4</sup>Percent of Target =  $\frac{\text{Actual Rate (pints product/A)}}{\text{Target Rate (pints product/A)}} \times 100$

ND01 and ND02 Plot 2 (60 fl oz product/A)

Table 10 Product Rate Determination (Continued)

Site Code And Plot	Date	App No	Volume		Treated Area (Acres/Linear ft)	Total product applied (oz) <sup>a</sup>	Target rate	Rate	
			Average Product applied per 50 feet	No of 50 feet sections				Actual Rate <sup>b</sup>	% of Target <sup>d</sup>
LA01 Plot 2	06/13/03	1	56.8 g	24	0.0689 A/ 1200 ft	48.0 oz/ 1200 ft row	40 oz/ 1000 ft row	40.0 oz/ 1000 ft row	100

<sup>a</sup>Total Product Applied = 1363.1 g x  $\frac{1 \text{ oz}}{28.4 \text{ g}}$  = 48.0 oz

<sup>b</sup>Actual Rate =  $\frac{48.0 \text{ oz}}{1200 \text{ ft row}} = \frac{X}{1000 \text{ ft row}}$  X =  $\frac{40.0 \text{ oz}}{1000 \text{ ft row}}$

Site Code And Plot	Date	App No	Volume			Total Pass Time (sec)	Calibrated Spray Rate (mL/sec)	Spray Mix Applied To Plot (mL) <sup>1</sup>	Treated Area (Acres)	Spray Rate (GPA) <sup>2</sup>	Rate	
			Test Substance (mL)	Carrier (mL)	Total Mixture (mL)						Actual Rate <sup>3</sup>	% of Target <sup>4</sup>
LA01 Plot 3	07/24/03	1	378	8620	8998	276.25	27.7	7652.1	0.0689	29.3	9.85 pt/A <sup>5</sup>	98.5

<sup>1</sup>Spray Mix Applied to Plot (mL) = Total Pass Times (sec) x Calibrated Spray Rate (mL/sec)

<sup>2</sup>Spray Rate (GPA) =  $\frac{\text{Spray Mix Applied to Plot (mL)}}{3785 \text{ mL/gal}} \times \frac{1}{\text{Treated Area (acres)}}$

<sup>3</sup>Disulfoton =  $\frac{\text{Test Substance in Spray Mixture (mL)}}{\text{Total Mixture Volume (mL)}} \times \text{Actual Spray Rate (GPA)} \times \frac{8 \text{ pt}}{1 \text{ gal}}$

<sup>4</sup>Percent of Target =  $\frac{\text{Actual Rate (pints product/A)}}{\text{Target Rate (pints product/A)}} \times 100$

LA01 Plot 2 (40 oz per 1000 ft of row)  
LA01 Plot 3 (10 pints/A)