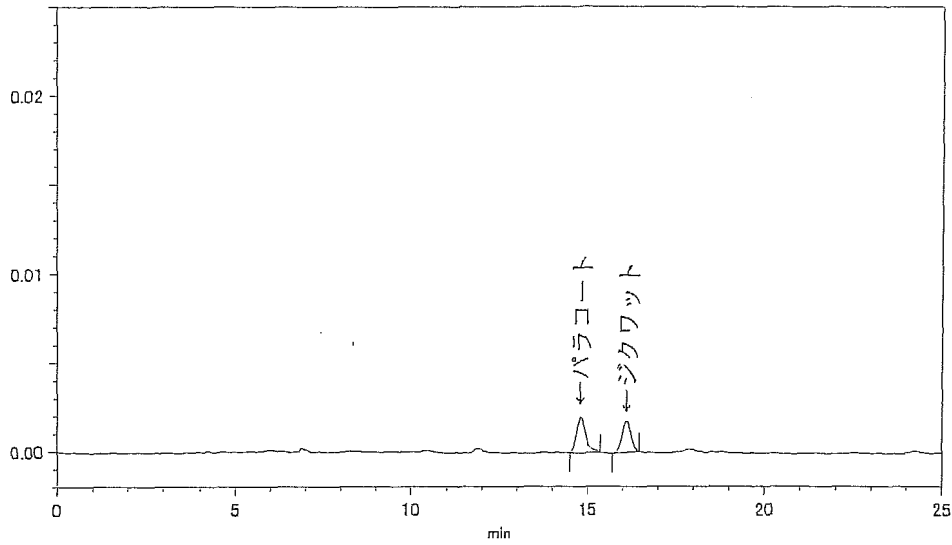


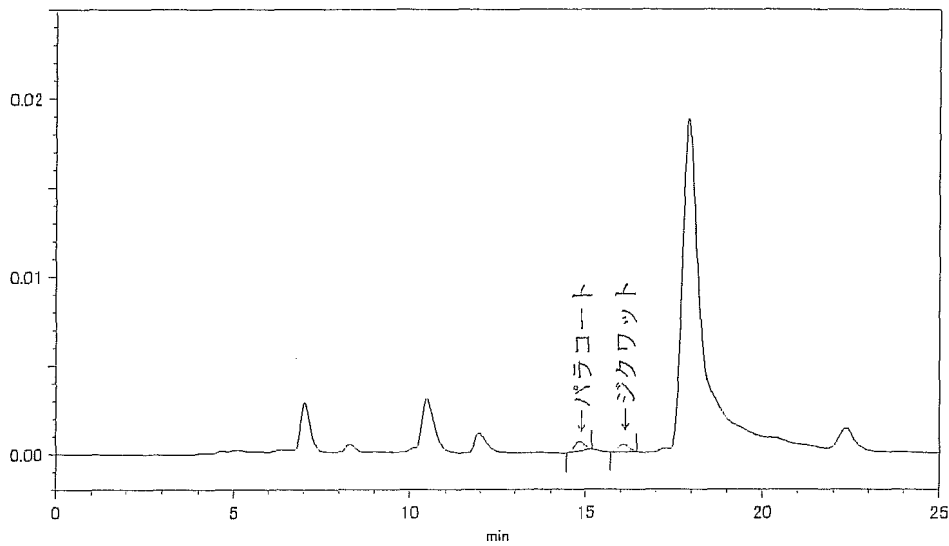
File name: D:\morimoto\m-data\DiquatParaquat\050218\R050218A-002



検出器 B  
(Ex:340nm,  
Em:430nm)

0.2 ppm 添加 (ジクワット+パラコート) 10  $\mu$ L/2 mL/0.5 g

File name: D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050324\B050324-007

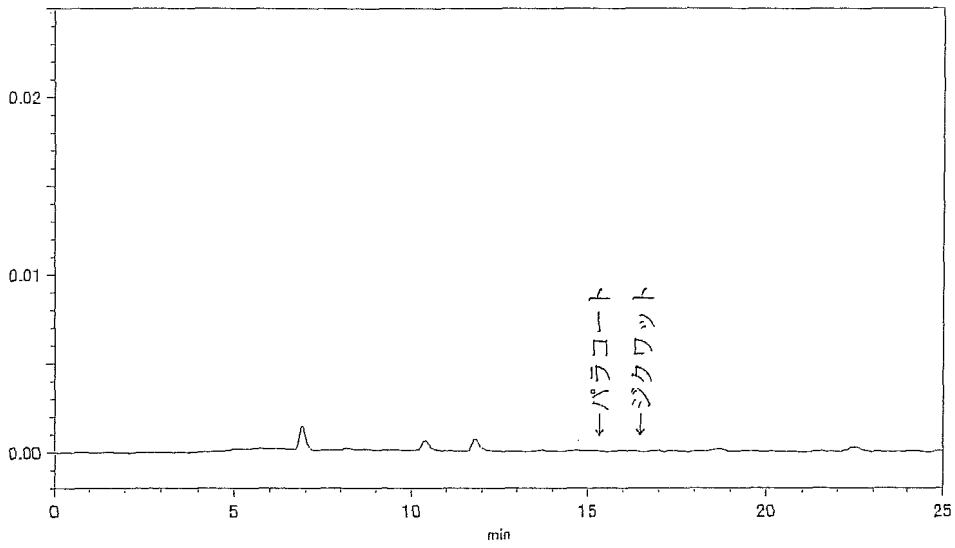


RF-10AXL  
(Ex:340nm,  
Em:430nm)

0.02 ppm 添加 (ジクワット+パラコート) 10  $\mu$ L/1 mL/0.5 g

図 7. ジクワットおよびパラコート (大豆試料) のクロマトグラム (続き)

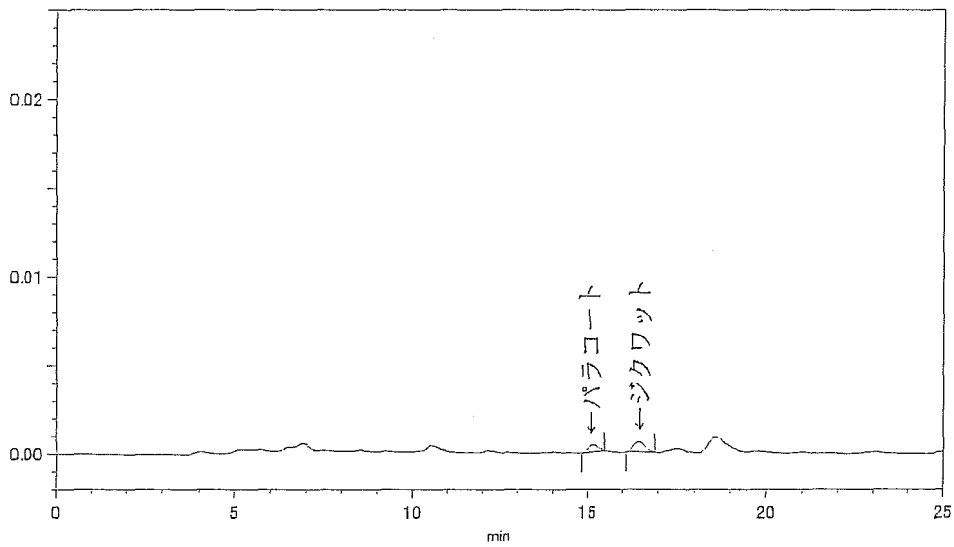
File name: D:\morimoto\m-data\DiquatParaquat\050125\050125-006



検出器 B  
(Ex:340nm,  
Em:430nm)

無処理(IA01-Plot11) 10  $\mu$ L/1 mL/0.5 g

File name: D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050407\B050407-007

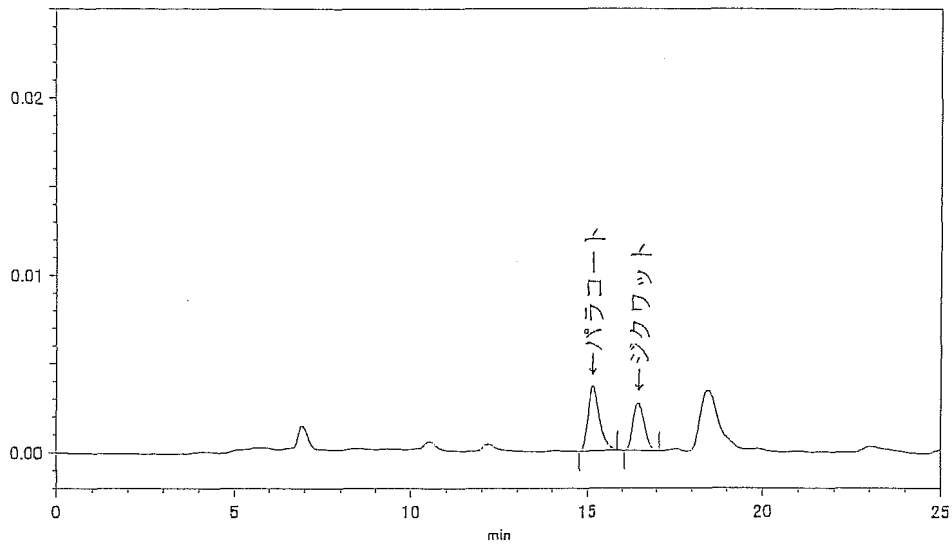


RF-10AXL  
(Ex:340nm,  
Em:430nm)

処理区(IA01-Plot12) 10  $\mu$ L/1 mL/0.5 g

図 7. ジクワットおよびパラコート (大豆試料) のクロマトグラム (続き)

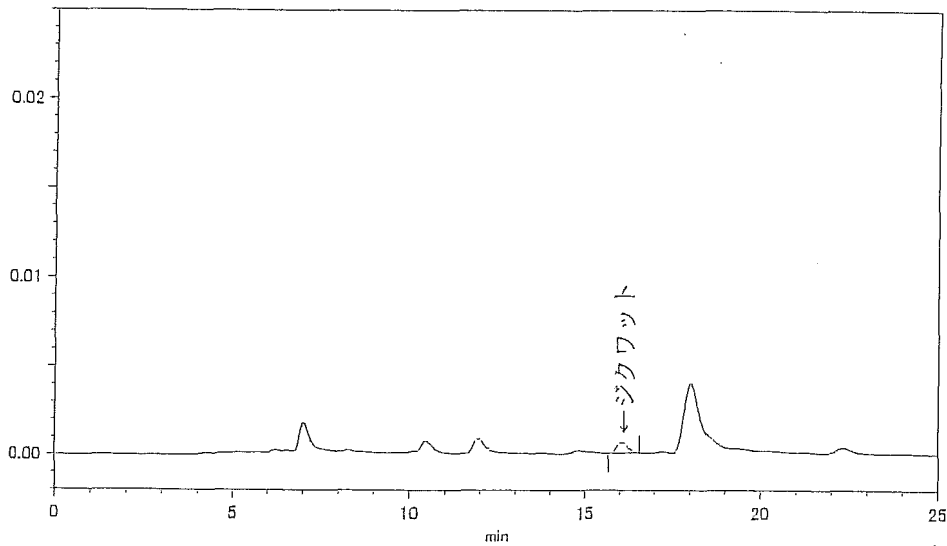
File name: D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050407\B050407-022



RF-10AXL  
(Ex:340nm,  
Em:430nm)

処理区(IA01-Plot13) 10  $\mu$ L/1 mL/0.5 g

File name: D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050323\B050323-007

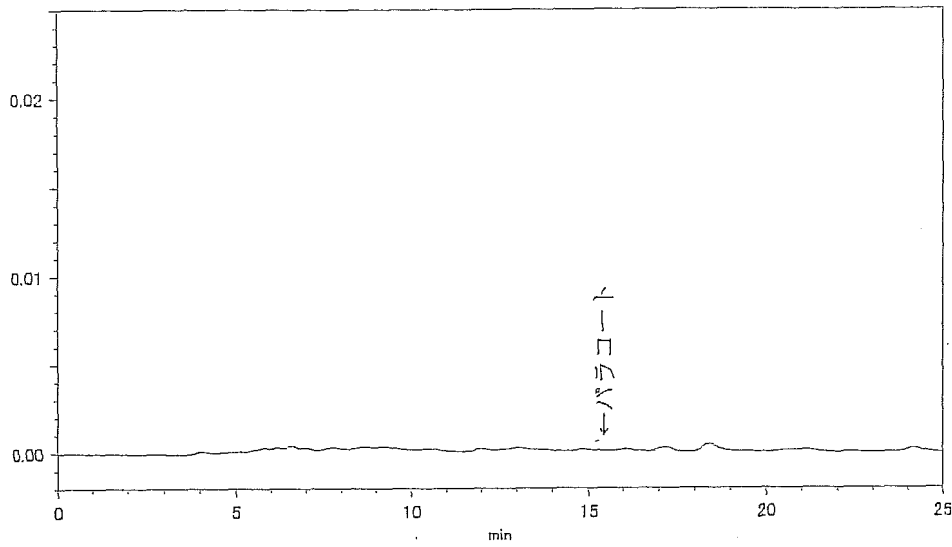


RF-10AXL  
(Ex:340nm,  
Em:430nm)

処理区(IA02-Plot20) 10  $\mu$ L/1 mL/0.5 g

図 7. ジクワットおよびパラコート (大豆試料) のクロマトグラム (続き)

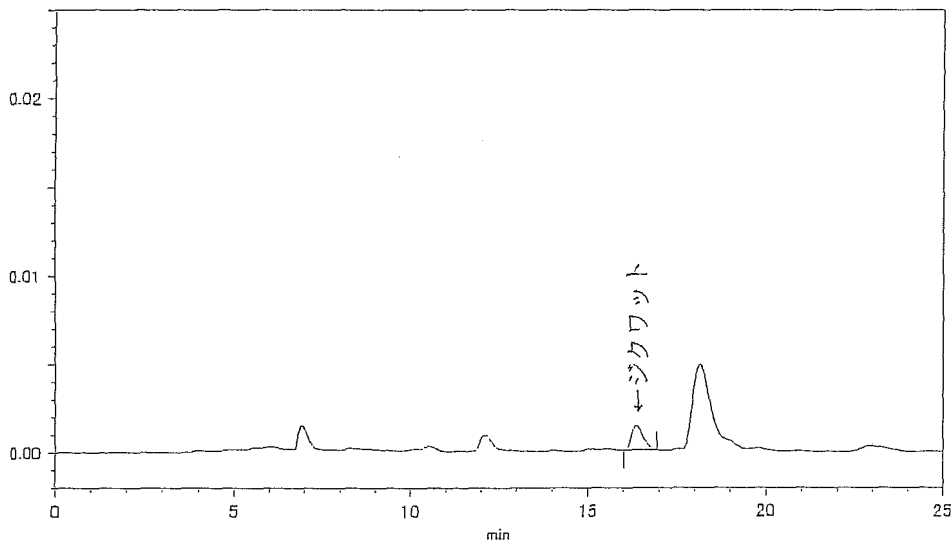
File name: D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050325\B050325-017



RF-10AXL  
(Ex:340nm,  
Em:430nm)

処理区(IA02-Plot21) 10  $\mu$ L/1 mL/0.5 g

File name: D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050426\A050426-004



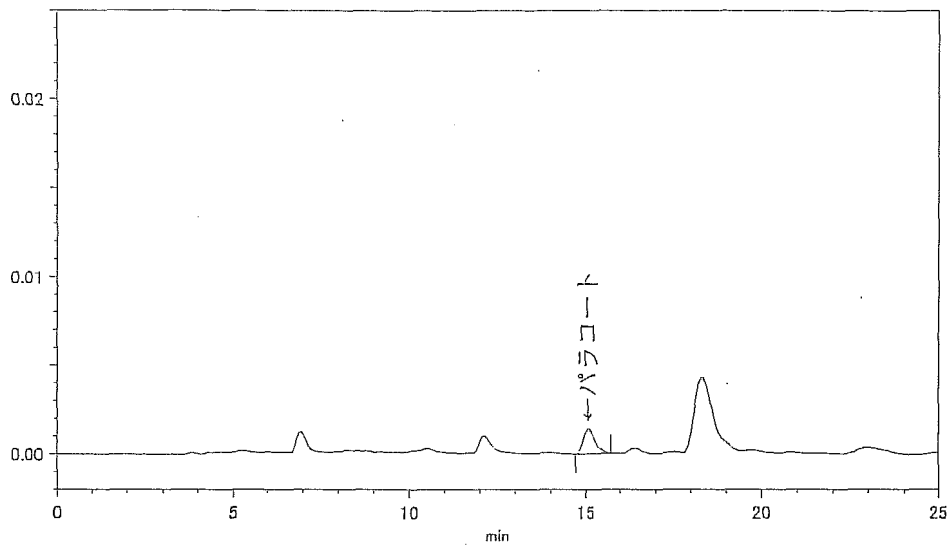
RF-10AXL  
(Ex:340nm,  
Em:430nm)

処理区(IA02-Plot22) 10  $\mu$ L/1 mL/0.5 g

図 7. ジクワットおよびパラコート (大豆試料) のクロマトグラム (続き)

File name:

D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050426\A050426-006

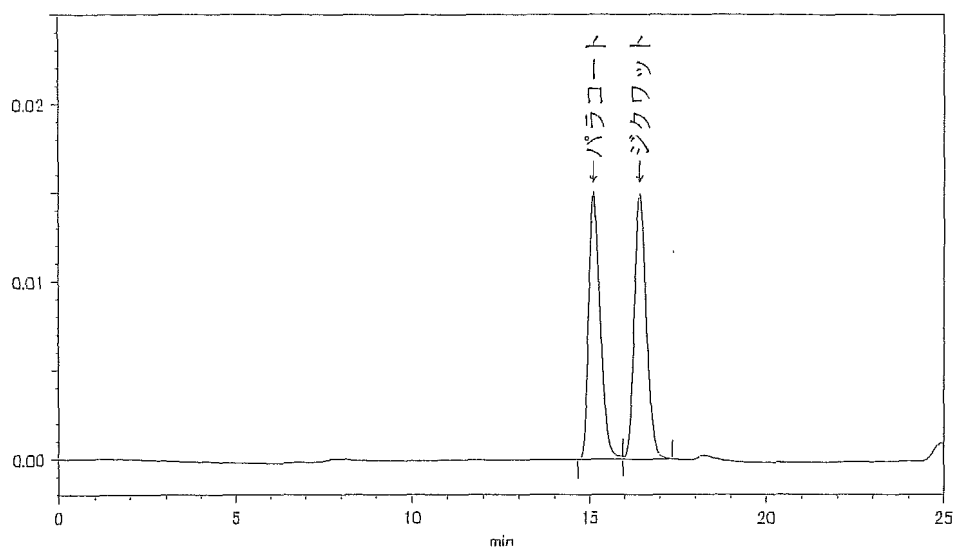


RF-10AXL  
(Ex:340nm,  
Em:430nm)

処理区(IA02-Plot23) 10  $\mu$ L/1 mL/0.5 g

図 7. ジクワットおよびパラコート (大豆試料) のクロマトグラム (続き)

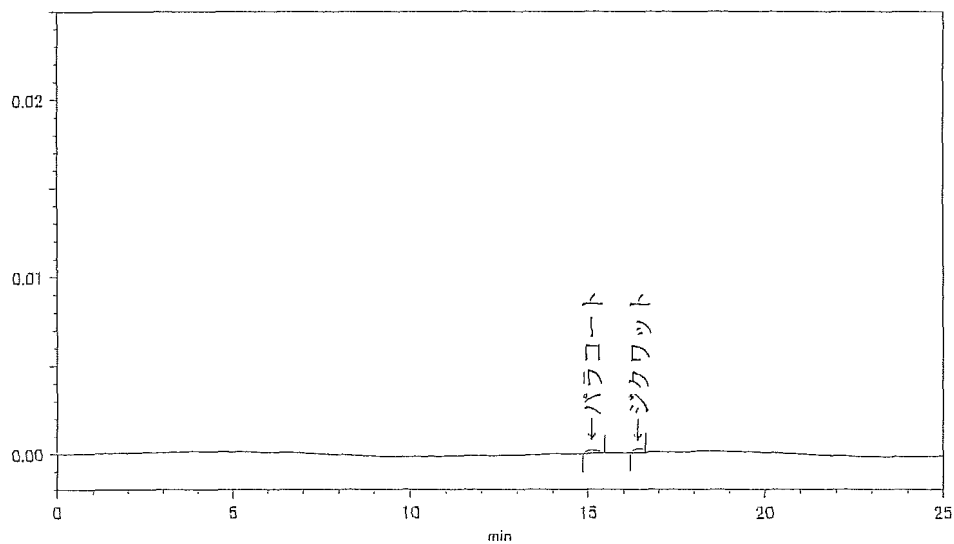
File name: D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050609\A050609-021



RF-10AXL  
(Ex:340nm,  
Em:430nm)

ジクワットおよびパラコート標準品 2 ng

File name: D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050609\A050609-016

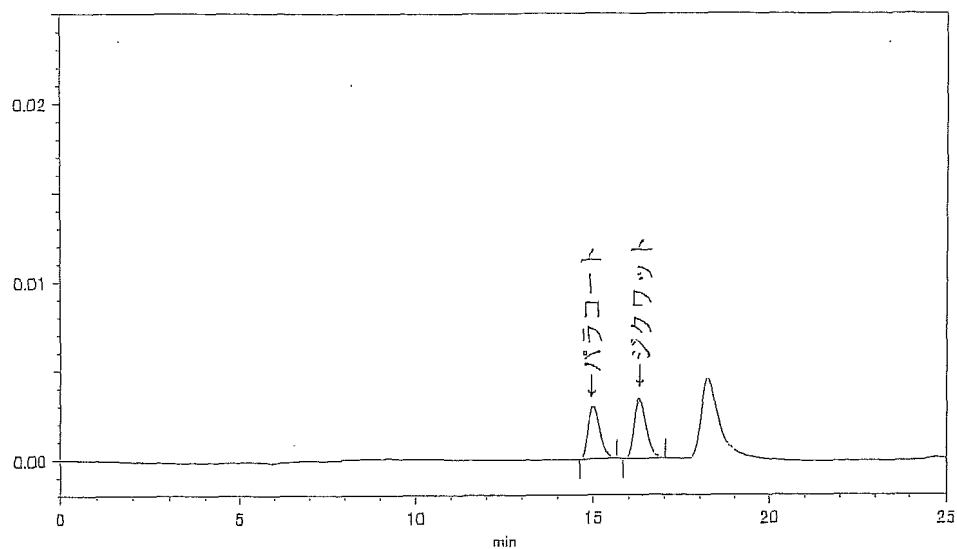


RF-10AXL  
(Ex:340nm,  
Em:430nm)

ジクワットおよびパラコート標準品 0.04ng

図 8. ジクワットおよびパラコート (玄麦試料) のクロマトグラム

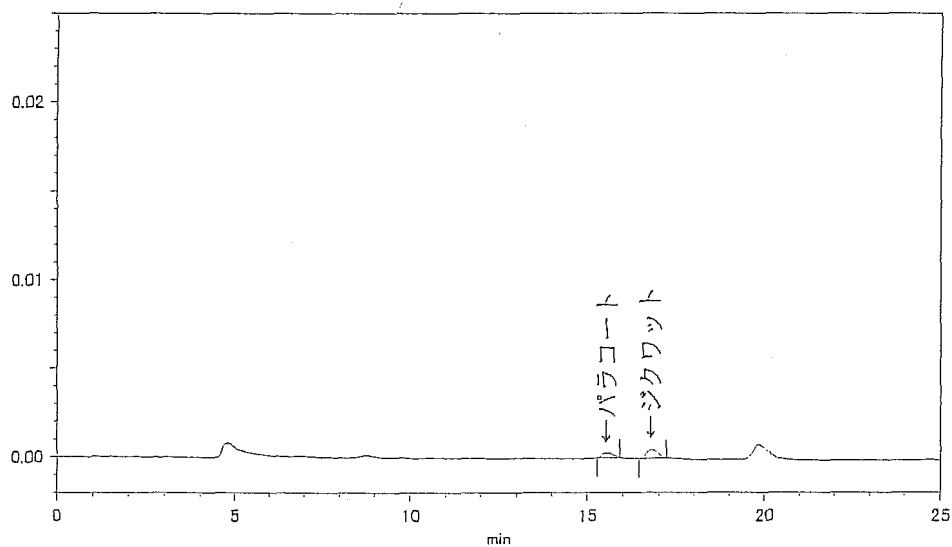
File name: D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050608\A050608-012



RF-10AXL  
(Ex:340nm,  
Em:430nm)

0.2 ppm 添加 10  $\mu$ L/1 mL/0.25 g

File name: D:\morimoto\m-data\DiquatParaquat\RES-LC-25\050822\050822-125



RF10AXL  
(Ex:340nm,  
Em:430nm)

0.04 ppm 添加 2  $\mu$ L/5 mL/10 g

図 8. ジクワットおよびパラコート (玄麦試料) のクロマトグラム (続き)

付表 1  
試料調製報告書



**STUDY TITLE**

Magnitude of the Residue of Several Test Compounds in  
Rice, Wheat, and Soybean Raw Agricultural Commodities

**STUDY NUMBER**

ERS24060

**DATA REQUIREMENT**

OPPTS 860.1000: Residue Chemistry Test Guidelines  
OPPTS 860.1500: Crop Field Trials

**AUTHORS**

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**FIELD REPORT COMPLETION DATE**

April 27, 2005

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## GOOD LABORATORY PRACTICE STATEMENT

The field phase reported herein, "Magnitude of the Residue of Several Test Compounds in Rice, Wheat, and Soybean Raw Agricultural Commodities," was conducted and reported following the 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.
- Acquisition and certification of test substances. All materials used were commercial products from various manufacturers.

This did not affect the integrity of the study.

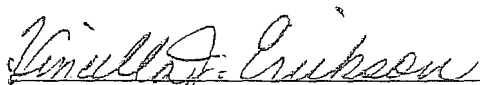
Author: Tim A. Cooley 27 Apr 05  
Tim A. Cooley Date  
Field Manager  
Excel Research Services, Inc.  
3021 West Dakota Avenue, Suite 110  
Fresno, CA 93722

## 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 April 18, 2005, 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 |
|-----------------|-------------------------|-----------------|--------------|---------------------------------|-----------------------------|
| AR01<br>Rice    | Protocol Review         | 07/18/04        | CC           | 07/19/04                        | 07/19/04                    |
|                 | Calibration/Application | 08/13/04        | CC           | 08/16/04                        | 08/16/04                    |
|                 | Sampling                | 10/01/04        | CC           | 10/06/04                        | 10/06/04                    |
|                 | Field Logbook           | 11/11/04        | CC           | 11/12/04                        | 11/12/04                    |
|                 | Field Logbook           | 02/02/05        | VJE          | 04/20/05                        | 04/20/05                    |
| ND01<br>Wheat   | Calibration/Application | 09/06/04        | SDK          | 09/24/04                        | 09/24/04                    |
|                 | Field Logbook           | 11/13/04        | SDK          | 11/16/04                        | 11/16/04                    |
|                 | Field Logbook           | 02/03/05        | VJE          | 04/20/05                        | 04/20/05                    |
| IA01<br>Soybean | Calibration/Application | 09/18/04        | PEJ          | 09/21/04                        | 09/21/04                    |
|                 | Field Logbook           | 11/12/04        | PEJ          | 11/16/04                        | 11/16/04                    |
|                 | Field Logbook           | 03/29/05        | VJE          | 04/20/05                        | 04/20/05                    |
| ND02<br>Wheat   | Field Logbook           | 04/16/05        | VJE          | 04/20/05                        | 04/20/05                    |
| IA02<br>Soybean | Field Logbook           | 11/12/04        | PEJ          | 11/16/04                        | 11/16/04                    |
|                 | Field Logbook           | 04/15/05        | VJE          | 04/20/05                        | 04/20/05                    |



Vincella J. Erickson  
Quality Assurance Officer  
A<sup>2</sup> Regulatory Professionals

27 Apr. 2005  
Date

Quality Assurance Personnel:

VJE = Vincella J. Erickson, A<sup>2</sup> Regulatory Professionals  
 CC = Cathy Caldwell, Mid-South Ag Research, 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 27 Apr 05  
Date  
Tim A. Cooley  
Field Manager  
Excel Research Services, Inc.  
3021 W. Dakota Avenue, Suite 110  
Fresno, CA 93722

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## I. SUMMARY

This magnitude of the residue study was conducted to provide raw agricultural commodity (RAC) samples of, and determine residue levels on, rice, wheat, and soybeans, following applications of Paraquat, Diquat, Carbaryl, Dimethoate, Methyl parathion, and Fenitrothion. Not all test substances were used on every crop or plot. For all test substances with multiple applications, the interval between applications was seven days. 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 will be done following GLP Standards in intent, but is not considered to be fully GLP compliant.

The field phase of this study was conducted from August 13, 2004, through October 25, 2004, corresponding to the first application through the last date of sample shipment. This study was conducted at five sites representing United States EPA Regions 4, 5, and 7, typical rice, wheat, and soybean-growing areas.

Weather conditions during the trial period were typical for the regions. If rainfall was not adequate for normal growth and development, irrigation was applied as needed. Agronomic practices, including fertilizer use and maintenance practices, were typical for rice, wheat, and soybean production at each site.

Rice, wheat, and soybean samples were successfully collected at all sites. Samples were stored frozen and shipped to Ibaraki, Japan, on dry ice via FedEx International Priority.

## II. INTRODUCTION

This study took place at five locations that represent typical rice, wheat, and soybean production conditions. The sites consisted of both untreated and treated plots. Treatment regimes consisted of both single and multiple applications at labeled and exaggerated rates and typical volume of carrier using commercial ground application equipment.

This study was conducted to provide raw agricultural commodity samples of rice, wheat, and soybean following applications of several test compounds. The test substances used were Paraquat, Diquat, Carbaryl, Dimethoate, Methyl parathion, and Fenitrothion. Not all test substances were used on every crop or plot. For all test substances with multiple applications, the interval between applications was seven days.

Each site contained a single untreated plot and multiple treated plots. Sites AR01 (rice), ND01 (wheat), IA01 (soybean), and IA02 (soybean) contained four treated plots and Site ND02 (wheat) contained two treated plots. Rice, wheat, or soybean samples were successfully collected at all sites.

This study is 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

A short description of each test substance follows:

- Paraquat and Diquat are contact herbicides used to control or suppress a broad spectrum of weeds by desiccating green plant tissue.
- Carbaryl is an insecticide for use on many crops.
- Dimethoate is a systemic insecticide-acaricide for a wide range of insects on many crops.
- Methyl parathion is an insecticide used to control many insects on many crops, including rice, soybean, and wheat.
- Fenitrothion is an insecticide and acaricide for use on cereals and many other crops.

The test substance product names, CAS Numbers (a.i.), lot numbers, appearance, dates test substances were obtained, amounts, and container sizes were recorded, if available, in the raw data by the principal field investigators.

## B. Test System

The test system was rice, wheat, and soybean grown in typical growing regions.

## C. Field Test Sites

### 1. Overview

The field phase of this study was comprised of five sites located in the major rice, wheat, and soybean-producing areas representing United States EPA Regions 4, 5, and 7.

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 rice, wheat, and soybean-growing regions. The site codes, locations, and Field Principal Investigators are listed in Table 1. A map of the United States listing the test site locations is presented in Figure 1.

Each site contained a single untreated plot and multiple treated plots. Sites AR01, ND01, IA01, and IA02 contained four treated plots and Site ND02 contained two treated plots. Rice, wheat, or soybean samples were successfully collected at each site.

### 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.

Soil types were loam at ND02 and silty clay loam at all other sites. Soil types were typical of the soils in the major rice, wheat, and soybean-growing areas representative of the United States EPA Regions 4, 5, and 7. Soil type information is presented in Table 3.

At Site AR01, the untreated and treated plots were 24 x 50 feet, except for Plot 5, which was cut to 12 x 50 feet for applications 2 and 3. At Site ND01, all plots were 40 x 70 feet; at Site ND02 all plots were 40 x 100 feet; and at Sites IA01 and IA02 all plots were 30 x 100 feet. At each site, the untreated plot was positioned at least



100 feet from the treated plot. The treated and untreated plots were identified by uniquely coded flags. Plot maps are presented in Figures 2 to 6. 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

The test substances used were Paraquat, Diquat, Carbaryl, Dimethoate, Methyl parathion, and Fenitrothion. Not all test substances were applied to every crop or plot. For all test substances with multiple applications, the interval between applications was seven days.

The test substances were applied at target rates as follows:

**Paraquat** was applied to soybeans once to Plot 12 (IA01) and Plot 21 (IA02) at the 1x rate (0.25 lb ai/A) and once to Plot 13 (IA01) and Plot 23 (IA02) at the 5x rate (1.25 lb ai/A). Actual application rates of paraquat on soybean ranged from 100 to 101 percent of target. Paraquat was applied at spray rates ranging from 22.2 to 22.9 gallons per acres (GPA).

**Diquat** was applied at the 1x rate (0.54 lb ai/A for rice and wheat and 0.5 lb ai/A for soybean) once to Plot 2 (AR01 rice), Plot 7 (ND01 wheat), Plot 12 (IA01 soybean), Plot 17 (ND02 wheat) and Plot 20 (IA02 soybean). Diquat was applied at the 5x rate (2.68 lb ai/A for rice and wheat and 2.5 lb ai/A for soybean) once to Plot 3 (AR01 rice), Plot 8 (ND01 wheat), Plot 13 (IA01 soybean), Plot 18 (ND02 wheat), and Plot 22 (IA02 soybean). Actual application rates of diquat on rice, wheat and soybean ranged from 100 to 104 percent of target. Diquat was applied at spray rates ranging from 15.5 to 24.4 GPA.

**Carbaryl** was applied to rice at the 1x rate (1.5 lb ai/A) twice to Plot 2 (AR01) and at the 5x rate (7.5 lb ai/A) twice to Plot 3 (AR01). The actual application rates of carbaryl on rice were 100 percent of target. Carbaryl was applied at spray rates of approximately 24 GPA.

Dimethoate was applied at the 1x rate (0.18 lb ai/A for rice, 0.375 lb ai/A for wheat, 0.5 lb ai/A for soybean) four times to Plot 4 (AR01 rice), twice to Plot 10 (ND01 wheat), and twice to Plot 15 (IA01 soybean). Dimethoate was applied at the 5x rate (0.89 lb ai/A for rice, 1.875 lb ai/A for wheat, and 2.5 lb ai/A for soybean) four times to Plot 5 (AR01 rice), twice to Plot 9 (ND01 wheat), and twice to Plot 14 (IA01 soybean). The actual application rates of dimethoate on rice, wheat, and soybean ranged from 97.1 to 102 percent of target. Dimethoate was applied at spray rates ranging from 19.4 to 28.2 GPA.

Methyl parathion was applied at the 1x rate (0.75 lb ai/A for rice and wheat and 1.0 lb ai/A for soybean) twice to Plot 4 (AR01 rice), Plot 10 (ND01 wheat), and Plot 15 (IA01 soybean). Methyl parathion was applied at the 5x rate (3.75 lb ai/A for rice and wheat, and 5.0 lb ai/A for soybean) twice to Plot 5 (AR01 rice), Plot 9 (ND01 wheat), and Plot 14 (IA01 soybean). The actual application rates of methyl parathion on rice, wheat, and soybean ranged from 99 to 102.9 percent of target. Methyl parathion was applied at spray rates ranging from 19.9 to 24.4 GPA.

Fenitrothion was applied at the 1x rate (1.63 pt/A for rice, 1.28 pt/A for wheat, 0.86 pt/A for soybean) three times to Plot 4 (AR01 rice), once to Plot 10 (ND01 wheat), and four times to Plot 15 (IA01 soybean). Fenitrothion was applied at the 5x rate (8.13 pt/A for rice, 6.42 pt/A for wheat, and 4.28 pt/A for soybean) three times to Plot 5 (AR01 rice), once to Plot 9 (ND01 wheat), and four times to Plot 14 (IA01 soybean). The actual application rates of fenitrothion on rice, wheat, and soybean ranged from 96 to 104 percent of target. Fenitrothion was applied at spray rates ranging from 109.8 to 114.5 GPA.

The actual application rates of all test substances on rice at Site AR01 ranged between 98.7 and 101 percent of target. Actual application rates of all test substances on wheat at Site ND01 ranged between 97.1 and 102.9 percent of target. Actual application rates of all substances on soybean at Site IA01 ranged between 96 and 104 percent of target. Actual application rates of all test substances on wheat at Site ND02 ranged between 103 and 104 percent of target. Actual application rates of all test substances on soybean at Site IA02 ranged between 100 and 101 percent of target.

Test substance shipping and storage information is presented in Table 7.

#### 4. Application Procedures

At all sites, the application equipment was calibrated prior to application of the test substance. All applications were verified by the time/volume technique, which is based on the output per time and equipment travel speed. The calibrations were conducted the same day as or the day before 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. Backpack sprayers and tractor-mounted boom sprayers were used to make the applications. 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 or on 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, rice and soybean samples were successfully collected. One sample was collected from each plot (one control and 2 to 4 treated plots) at each of the five sites. In all cases, the untreated plots were sampled before the treated plots. Equipment was thoroughly cleaned before use and between treated samples. Combine-type equipment was flushed with untreated grain at all sites except Site AR01 where the combine was flushed with plot grain before beginning the harvest of the trial site plots. Samples were placed in pre-labeled plastic-lined sample bags. If sample material was touched by person(s) collecting sample, disposal gloves were used and changed between treated samples.

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

#### D. Deviations

Deviations that occurred during the field phase of this study were documented and reported to the Study Director. The deviations are listed numerically and had no impact on the results of this study.

##### Deviation No. 1

At Site IA02, the applications to Plots 21 and 23 were made at 16 days before harvest and not the protocol-requested 15 days before harvest.

##### Deviation No. 2

At Site AR01, samples were shipped with husks on the rice. The protocol sample list shows husked rice.

##### Deviation No. 3

At Site ND01, Plots 9 and 10, the interval between Dimethoate Applications 1 and 2 was 6 days instead of the protocol-requested 7-day interval.

#### 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.