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Biochemistry Department
Ciba-Geigy Corporation
Ciba Plant Protection
Greensboro, North Carolina

STORAGE STABILITY OF TOTAL RESIDUES OF METALAXYL IN
WEATHERED CROPS UNDER FREEZER STORAGE CONDITIONS

Report No.: ABR-93009 Project No.: 409986
Study No.: 8-90

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STATEMENT CONCERNING GOOD LABORATORY PRACTICES

This study was conducted in accordance with the applicable EPA Good Laboratory Practice Standards (40 CFR Part 160) with the following exceptions:

1. The biological phase of the study was initiated prior to and completed after the effective date of the applicable GLP regulations (October 16, 1989).
2. In accordance with 40 CFR Part 160, Subpart F, 160.113, tank mix stability was not determined for any of the test substance treatments in any of the projects from which samples were obtained for this storage stability study.
3. Metalaxyl standards obtained from the Production Technical Analytical Services (PTAS) Department were stored both in a freezer and at room temperature. PTAS instructions call for storage at room temperature. Storage at freezer temperatures is not believed to have affected the integrity of the standard(s).

5.1.2.e Woo

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8-27-93
Date

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Quality Assurance Unit

QUALITY ASSURANCE STATEMENT

Study Title: STORAGE STABILITY OF TOTAL RESIDUES OF METALAXYL IN WEATHERED CROPS UNDER FREEZER STORAGE CONDITIONS

Study Number: 8-90

Project Number: 409986

ABR Number: ABR-93009

Pursuant to Good Laboratory Practice Regulations, this statement verifies that the aforementioned study was inspected and/or audited and the findings reported to management and to the Study Director by the Quality Assurance Unit on the dates listed below:

<u>AUDIT TYPE</u>	<u>INSPECTION/AUDIT DATE</u>	<u>REPORTING DATE</u>
Protocol Audit	1/26/90	1/26/90
In-Progress Inspection	11/16/90	12/10/90
In-Progress Inspection	4/10/91	4/12/91
ABR Audit	4/28-29, 5/3-6/93	5/9/93

Prepared By

5.1.2.e Woo

Date: 5-6-93

GENERAL INFORMATION

Study Participants:

Study Director: 5.1.2.e Woo (6/26/92 - present)

Technical Personnel: 5.1.2.e Woo (study director
(1/30/90 - 6/26/92)

5.1.2.e Woo

Refer to Testing Facilities
below for field personnel.

CIBA-GEIGY Code: CGA-48988
Generic Name: Metalaxyl
(See Table I for details.)

Protocol Number: 8-90 and Amendments 1-5

Testing Facilities: Biological Phase

Cranberry samples

Field trial: NE-FR-401-89
Location: Massachusetts
Field Rep: 5.1.2.e Woo
Address: Northeast Regional
Office
Ciba-Geigy Corp.
1200 Washington Rd.
Washington, PA 15301
Phone: 412-5.1.2.e Woo

Pepper samples

Field trial: OS-FR-605-89
Location: North Carolina
Field Rep: 5.1.2.e Woo
Address: 5605 Knollwood Dr.
Raleigh, NC 27609
Phone: 919-5.1.2.e Woo

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I. SUMMARY/INTRODUCTION

Introduction

Metalaxyl* N-(2,6-dimethylphenyl)-N-(methoxyacetyl)alanine methyl ester, developed under CIBA Code Number CGA-48988, is a systemic fungicide specific for the Oomycete class of fungi. Metalaxyl is registered for use on a wide variety of crops.

Previously, Ciba submitted storage stability data from two studies, ABR-80028¹ (MRID No. 40534802) and ABR-86044² (MRID No. 40106691; also submitted as part of PP 6F3387/FAP 6H5499). In the first study, the stability of parent metalaxyl was shown in fortified potatoes and tobacco for 12 months and the stability of total residues of metalaxyl was shown in field-treated samples of potatoes and tobacco after 18 months of freezer storage (-15°C). The freezer stability of parent metalaxyl and five metabolites individually fortified on strawberries, apples, cabbage, lettuce, and potatoes was reported in the second study; freezer storage stability was demonstrated for all compounds for up to 12 months.

The objectives of this study were 1) to provide data on the stability of residues of metalaxyl in weathered field samples of cranberries, peppers, potatoes, and spinach samples stored under freezer conditions for 18-24 months and 2) to satisfy the requirements of the Final Registration Standard Tolerance Reassessment (FRSTR) of September 23, 1988. Additional results from the analysis of samples stored for approximately 38 months will be presented in a separate report (amendment to ABR-93009).

Summary

Weathered samples of peppers, potatoes, and spinach were stored for 24 months under freezer

*Refer to Figure 1 for the structures of metalaxyl and 2,6-dimethylaniline (DMA).

storage conditions (approximately -20°C). Weathered samples of cranberries were stored under identical conditions for 18 months. These samples were analyzed periodically for total residues of metalaxyl determined as 2,6-dimethylaniline (DMA). Results show that total residues of metalaxyl, i.e., combined parent and metabolites containing the DMA moiety and N-[2-(hydroxymethyl)-6-methylphenyl]-N-(methoxyacetyl) alanine methyl ester, are stable in peppers, potatoes, and spinach for at least 24 months and in cranberries for at least 18 months under freezer storage conditions.

II. MATERIALS

Test Substances

Analytical standards of 2,6-dimethylaniline and metalaxyl were obtained from the Production Technical Analytical Services (PTAS) Department, Ciba Plant Protection, Greensboro, North Carolina. Additional information concerning these standards can be found in Table I.

The formulations employed in this study were Ridomil 2E, Ridomil 5G, and Ridomil MZ58. Refer to the individual Residue Test Reports³⁻⁶ for additional information regarding the formulations, treatment rates, etc.

Test Commodities

The identification (sample code) and source of control and treated (weathered) samples of cranberries, peppers, potatoes, and spinach are described in Table II.

III. METHODS

Experimental Design

The experimental design is described in detail in Residue Chemistry Department Protocol 8-90⁷. Field samples were collected and shipped frozen to Ciba-Geigy Corporation, Greensboro. Upon arrival, samples were stored at approximately -20°C with

the temperature monitored continuously. Sample preparation was performed according to the Ciba SOP 7.21, Revision 0⁸. After preparation, the samples were stored in polyethylene bags or bottles. On the day of analysis, weathered crop samples were removed from the freezer, subsamples were taken, and samples were returned to the freezer within eight hours. Each sample set consisted of a control sample, 2 recoveries (control samples fortified with parent metalaxyl immediately before extraction), and 2 weathered crop samples (replicates of the same sample).

As documented by Amendment 3 to Protocol 8-90, this study was extended from two years to approximately 38 months. The reason for extending this storage stability study beyond two years is because the longest interval of freezer storage for all metalaxyl/crop samples is approximately 38 months. Sample storage interval data is summarized in ABR-90016⁹ and Amendment 1¹⁰ to ABR-90016.

Analytical Method

Analytical method AG-395¹¹ (P.A.M. enforcement method) was employed for the analysis of total residues of metalaxyl in weathered crop samples. Several modifications to AG-395 were employed. Crop samples are extracted by refluxing with 80% (v/v) methanol/water for two hours as described in Section 5.4, "Extraction - Dry Crop." This extraction has been found to be more efficient than the polytron extraction (Section 5.3). In Section 5.5.4, the water and sodium hydroxide were doubled in volume. In Sections 5.7.6 and 5.7.7, the trifluoroacetic acid addition step for the formation of DMA-TFA was found to be unnecessary and was omitted. DMA residues in dichloromethane from Section 5.7.5 were concentrated for GC analysis.

Analytical standards of metalaxyl used for fortification purposes were prepared per AG-395. DMA standards for gas chromatographic analysis were prepared in dichloromethane. A Hewlett-Packard 5880 gas chromatograph with a NPD detector

operating in the Nitrogen-specific mode was used for analysis. A 30 m x 0.32 mm I.D. DB-WAX capillary column was employed for separation. GC oven program parameters were modified to improve separation. The GC oven program employed for most analyses is listed as follows: initial temperature (60°C); initial time (1.0 min); program rate 1 (4.0°C/min); final temperature 1 (150°C); program rate 2 (25°C/min); final temperature 2 (230°C); final time 2 (3 min). The injection port and detector were set at 225°C and 300°C, respectively. Any change to these parameters is documented in the raw data.

DMA results calculated in parts-per-million (ng DMA found/mg crop injected) are converted to metalaxyl equivalents using the factor 2.308. Calculations are described in AG-395 and SOP 8.3, Revision 1¹². Examples of analysis report sheets which show all of the intermediate results from each calculation are included in the Residue Test Reports. These analysis report sheets correspond to the representative figures shown in this report.

IV. RESULTS/DICUSSION

Residue Results

Sample preparation, fortification, extraction, and analysis dates are presented in Table III. Results of analyses to determine total residues of metalaxyl in weathered samples of cranberries, peppers, potatoes, and spinach are presented in Tables IV, V, VI, and VII, respectively. Representative chromatograms of standard, control, recovery and weathered samples of cranberries, peppers, potatoes, and spinach are shown in Figures 2, 3, 4, and 5, respectively. Representative chromatograms of DMA standards (range 0.02-0.40 ng) are presented in Figure 6. The corresponding calibration curve of peak heights versus amounts of DMA is shown in Figure 7. Plots of total residues of metalaxyl determined in cranberries, peppers, potatoes, and spinach versus freezer storage time (days) are presented in Figures 8, 9, 10, and 11,

respectively. [Note: the average ppm of replicate analyses was calculated and plotted versus time.]

One of residues determined in peppers at 201 days was 0.16 ppm, which was significantly lower than the corresponding replicate analysis (0.38 ppm) or any of the other residue values determined over two years. All of the other residues of metalaxyl determined in peppers were in the range 0.47-0.61 ppm. The 0.16 ppm residue is believed to be an outlier. No significant loss of total residues of metalaxyl in peppers is believed to have occurred over two years of freezer storage.

Overall, total residues of metalaxyl determined as DMA are stable in peppers, potatoes, and spinach for up to 24 months and in cranberries for up to 18 months when these crops are stored under freezer conditions (-20°C).

Method Recovery Validation Data

The validation of Analytical Method AG-395 has been demonstrated in ABR-83033¹³. Results of procedural recoveries of metalaxyl from control samples of cranberries, peppers, potatoes, and spinach fortified prior to extraction are presented in Tables VIII, IX, X, and XI, respectively.

Circumstances Affecting Study

This study was inadvertently assigned under two project numbers, 409925 and 409986. The final report and raw data are archived under project number 409925, study number 8-90.

Over the duration of the study, metalaxyl standards obtained from PTAS were stored both in a freezer and at room temperature. PTAS instructions call for storage at room temperature. Storage at freezer temperatures is not believed to have affected the integrity of the metalaxyl standards.

V. CONCLUSION

Total residues of metalaxyl determined as 2,6-dimethylaniline are stable in peppers, potatoes, and spinach samples stored for up to 24 months and in cranberries stored for up to 18 months at -20°C. Results of analyses of these substrates at 38 months of freezer storage will be submitted as an amendment to this ABR at a later date.

Cranberries, peppers, potatoes, and spinach are representative commodities of small fruits and berries, fruiting vegetable, root and tuber vegetable, and leafy vegetable crop groups, respectively. Additional crops and crop groups were represented in two previous storage stability studies reported in ABR-80028 and ABR-86044. The data reported herein and the data reported in the two previous studies were consistent - total residues of metalaxyl were stable in all crops stored under freezer conditions for storage times ranging from 12 to 24 months. Assuming that results from the analysis of the 38-month storage interval further confirm the long-term freezer stability of metalaxyl residues in the four crops involved in the current study, we believe that, taken as a whole, the storage stability data base for this compound will be adequate for translation to all other crops for which metalaxyl has current or pending tolerances.

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VI. TABLES AND FIGURES

TABLE I: ANALYTICAL STANDARDS

Metalaxyl

N-(2,6-dimethylphenyl)-N-(methoxyacetyl)alanine methyl ester

Ciba Code Number: CGA-48988

CAS Registry Number: 57837-19-1

Storage Conditions: Room Temperature/Freezer

[Note: during the duration of the study, metalaxyl standards obtained from PTAS were stored both in a freezer and at room temperature before preparation of GC and fortification standards.]

2,6-Dimethylaniline (DMA)

Ciba Code Number: CGA-72649

CAS Registry Number: 87-62-7

Storage Conditions: Freezer

Interval (days)	Compound	Code Number	Purity %	Dept. ID #	Reassay Date
<u>Cranberries</u>					
0,	Metalaxyl	S87-1208	95.8	B05692	5/93
	DMA	39254	99.0	B05693	8/91
186	Metalaxyl	S87-1208	95.8	B06192	5/93
	DMA	39254	99.0	B06191	1/92
409,	Metalaxyl	S87-1208	95.8	B06619	5/93
	DMA	39254	99.0	B06620	9/92
<u>Peppers</u>					
0,	Metalaxyl	S87-1208	95.8	B05692	5/93
	DMA	39254	99.0	B05691	1/91
201,	Metalaxyl	S87-1208	95.8	B05962	5/93
	DMA	39254	99.0	B05963	8/91
376,	Metalaxyl	S87-1208	95.8	B06192	5/93
	DMA	39254	99.0	B06191	1/92
767	Metalaxyl	S87-1208	95.8	B06619	5/93
	DMA	39254	99.0	B06620	9/92

TABLE III. FORTIFICATION, EXTRACTION, AND ANALYSIS DATES FOR CRANBERRY, PEPPER, POTATO, AND SPINACH SAMPLES

<u>Storage Interval (Days)</u>	<u>Storage Date</u>	<u>Extraction Date</u>	<u>Analysis Date</u>
<u>Cranberries</u>			
0	10/12/90	10/12/90	11/12/90
35	10/12/90	11/16/90	11/20/90
186	10/12/90	04/16/91	04/20/91
409	10/12/90	11/25/91	12/02/91
522	10/12/90	03/17/92	03/20/92
<u>Peppers</u>			
0	02/09/90	02/09/90	02/14/90
59	02/09/90	04/09/90	04/11/90
201	02/09/90	08/29/90	09/10/90
278	02/09/90	11/14/90	11/19/90
376	02/09/90	02/20/91	02/22/91
566	02/09/90	08/29/91	09/09/91
767	02/09/90	03/17/92	03/23/92
<u>Potatoes</u>			
0	03/29/90	03/29/90*	04/11/90
196	03/29/90	10/12/90	10/31/90
377	03/29/90	04/11/91	04/17/91
605	03/29/90	11/25/91	12/02/91
718	03/29/90	03/17/92	03/19/92
<u>Spinach</u>			
0	01/31/90	01/31/90	02/09/90
51	01/31/90	03/23/90	04/02/90
253	01/31/90	10/11/90	11/12/90
384	01/31/90	02/19/91	02/21/91
568	01/31/90	08/22/91	08/29/91
729	01/31/90	01/30/92	02/04/92

*The control sample was lost during workup; it was reextracted on 04/04/90.

TABLE IV. RESULTS FROM THE ANALYSIS OF WEATHERED CRANBERRY SAMPLES

<u>Storage Interval (Days)</u>	<u>Sample Code</u>	<u>Total Residues of Metalaxyl (PPM) *</u>
0	0-C-C	<0.05
	0-W-1-C	0.47
	0-W-2-C	0.42
35	1-C-C	<0.05
	1-W-1-C	0.75
	1-W-2-C	0.51
186	6-C-C	<0.05
	6-W-1-C	0.54
	6-W-2-C	0.54
409	12-C-C	<0.05
	12-W-1-C	0.54
	12-W-2-C	0.46
522	18-C-C	<0.05
	18-W-1-C	0.66
	18-W-2-C	0.60

*Detected as 2,6-dimethylaniline and reported as metalaxyl equivalents using the conversion factor 2.308.

Note: Residue results are not corrected for control values.
Residue results are corrected for average procedural recoveries <100%.
Procedural recoveries are corrected for control values.

Sample Code:

Interval - Sample Type - Replicate - Substrate

C = Control
R = Recovery
W = Weathered

C = Cranberries

TABLE V. RESULTS FROM THE ANALYSIS OF WEATHERED PEPPER SAMPLES

Storage Interval (Days)	Sample Code	Total Residues of Metalaxyl (PPM)*
0	0-C-Pr	0.05
	0-W-1-Pr	0.48
	0-W-2-Pr	0.48
59	2-C-Pr	0.05
	2-W-1-Pr	0.57
	2-W-2-Pr	0.56
201	6-C-Pr	0.05
	6-W-1-Pr	0.16
	6-W-2-Pr	0.38
278	9-C-Pr	0.05
	9-W-1-Pr	0.52
	9-W-2-Pr	0.55
376	12-C-Pr	0.06
	12-W-1-Pr	0.49
	12-W-2-Pr	0.47
566	18-C-Pr	0.06
	18-W-1-Pr	0.57
	18-W-2-Pr	0.61
767	24-C-Pr	0.05
	24-W-1-Pr	0.49
	24-W-2-Pr	0.54

*Detected as 2,6-dimethylaniline and reported as metalaxyl equivalents using the conversion factor 2.308.

Note: Residue results are not corrected for control values.
Residue results are corrected for average procedural recoveries <100%.
Procedural recoveries are corrected for control values.

Sample Code:

Interval - Sample Type - Replicate - Substrate

C = Control
R = Recovery
W = Weathered

Pr = Peppers

TABLE VI. RESULTS FROM THE ANALYSIS OF WEATHERED POTATO SAMPLES

<u>Storage Interval (Days)</u>	<u>Sample Code</u>	<u>Total Residues of Metalaxyl (PPM)*</u>
0	0-C-Po	<0.05
	0-W-1-Po	0.22
	0-W-2-Po	0.25
196	6-C-Po	<0.05
	6-W-1-Po	0.16
	6-W-2-Po	0.18
377	12-C-Po	<0.05
	12-W-1-Po	0.19
	12-W-2-Po	0.20
605	18-C-Po	<0.05
	18-W-1-Po	0.15
	18-W-2-Po	0.18
718	24-C-Po	<0.05
	24-W-1-Po	0.25
	24-W-2-Po	0.23

*Detected as 2,6-dimethylaniline and reported as metalaxyl equivalents using the conversion factor 2.308.

Note: Residue results are not corrected for control values.
Residue results are corrected for average procedural recoveries <100%.
Procedural recoveries are corrected for control values.

Sample Code:

Interval - Sample Type - Replicate - Substrate

C = Control
R = Recovery
W = Weathered

Po - Potatoes

TABLE IX. PROCEDURAL RECOVERIES OF METALAXYL DETERMINED AS DMA FROM FORTIFIED CONTROLS OF PEPPERS

<u>Storage Interval (Days)</u>	<u>Sample Code</u>	<u>PPM Added</u>	<u>Percent Recovery</u>
0	0-R-1-Pr	0.05	91
	0-R-2-Pr	4.0	89
59	2-R-1-Pr	0.05	50
	2-R-2-Pr	1.0	76
201	6-R-1-Pr	0.05	154
	6-R-2-Pr	1.0	87
278	9-R-1-Pr	0.05	60
	9-R-2-Pr	1.0	74
376	12-R-1-Pr	0.05	96
	12-R-2-Pr	1.0	94
566	18-R-1-Pr	0.05	60
	18-R-2-Pr	1.0	95
767	24-R-1-Pr	0.05	62
	24-R-2-Pr	1.0	82
	mean		83.6
	std. dev.		25.3
	n		14
	range		50-154

Procedural recoveries are corrected for control values.

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TABLE XI. PROCEDURAL RECOVERIES OF METALAXYL DETERMINED AS DMA FROM FORTIFIED CONTROLS OF SPINACH

<u>Storage Interval (Days)</u>	<u>Sample Code</u>	<u>PPM Added</u>	<u>Percent Recovery</u>
0	0-R-1-S	0.05	122
	0-R-2-S	2.0	86
51	2-R-1-S*	0.05	77
253	8-R-1-S	0.05	58
	8-R-2-S	2.0	84
384	12-R-1-S	0.05	64
568	18-R-1-S	0.05	65
	18-R-2-S	3.0	91
729	24-R-1-S	0.05	67
	24-R-2-S	3.0	77
	mean		79.1
	std. dev.		18.5
	n		10
	range		58-122

Procedural recoveries are corrected for control values.

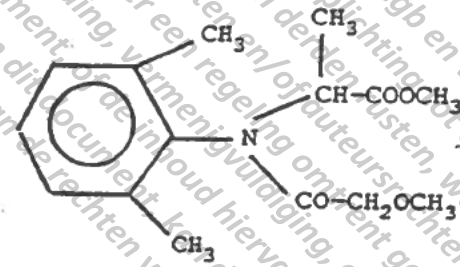
*The control sample (1-1-A) from Field Trial 02-FR-003-89 was used for analysis.

FIGURE 1. CHEMICAL NAMES AND STRUCTURES

Metalaxyl
CGA-48988

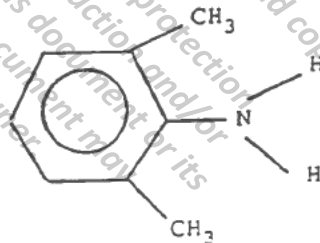
N-(2,6-dimethylphenyl)-N-(methoxyacetyl)alanine methyl ester

$C_{15}H_{21}NO_4$



2,6-Dimethylaniline (DMA)
CGA-72649

$C_8H_{11}N$

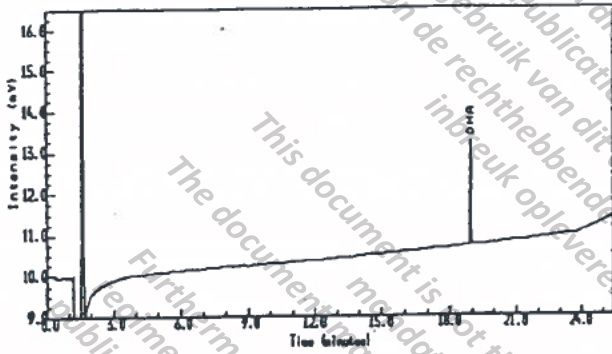


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FIGURE 2. REPRESENTATIVE CHROMATOGRAMS FROM
THE ANALYSIS OF CRANBERRIES
(Storage Interval 409 Days)

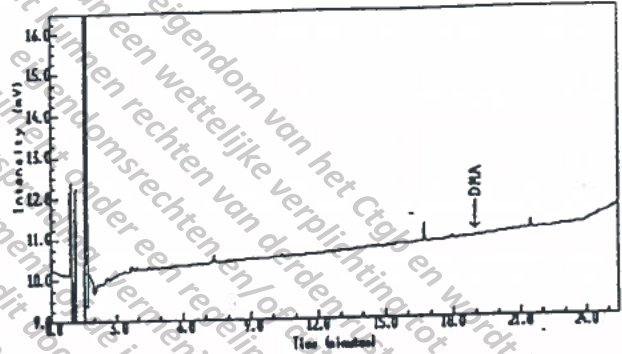
Acquired on 3-DEC-1991 at 02:51



C-G Multichrom 1.8 (BONES)

1

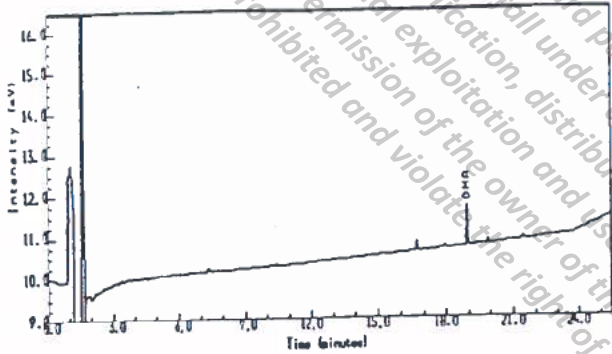
Acquired on 2-DEC-1991 at 23:20



C-G Multichrom 1.8 (BONES)

2

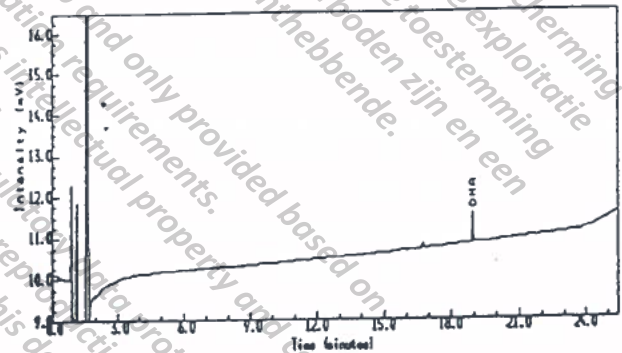
Acquired on 3-DEC-1991 at 03:26



C-G Multichrom 1.8 (BONES)

3

Acquired on 3-DEC-1991 at 01:05



C-G Multichrom 1.8 (BONES)

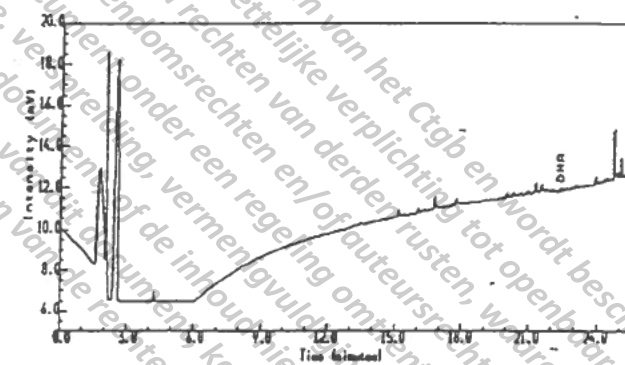
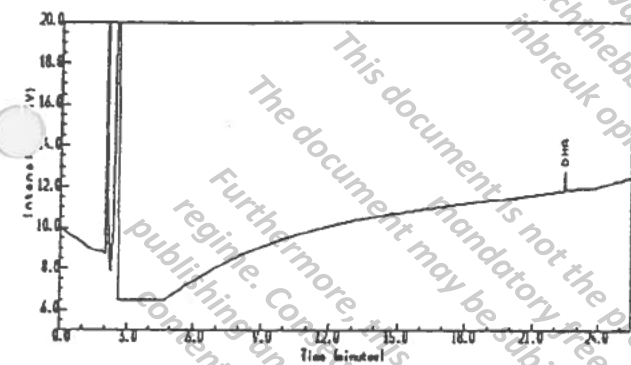
4

1. standard, DMA, 0.40 ng
2. 1-1-A, control, 1.838 mg injected, (0.00 ng) <0.02 ng found, (0.00 ppm) <0.05ppm
3. 1-1-A + 1.0 ppm, 0.368 mg injected, 0.115 ng found, 0.724 ppm found, 0.724 ppm corrected for control, 72% recovery
4. 2-1-B, treated sample, 0.919 mg injected, 0.152 ng found, 0.46 ppm

FIGURE 3. REPRESENTATIVE CHROMATOGRAMS FROM THE ANALYSIS OF PEPPERS (Storage Interval 566 Days)

Acquired on 9-SEP-1991 at 15:52

Acquired on 9-SEP-1991 at 13:13



C-G Multichrom 1.8 (BONES)

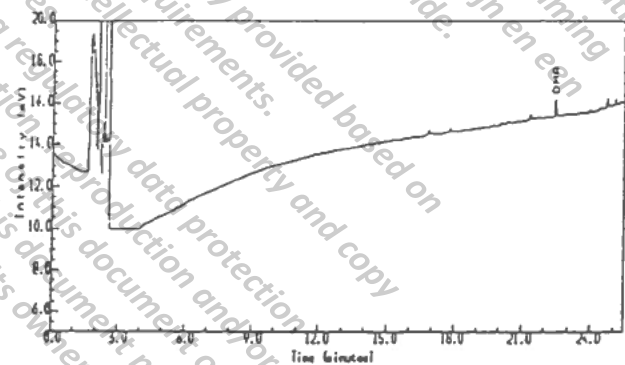
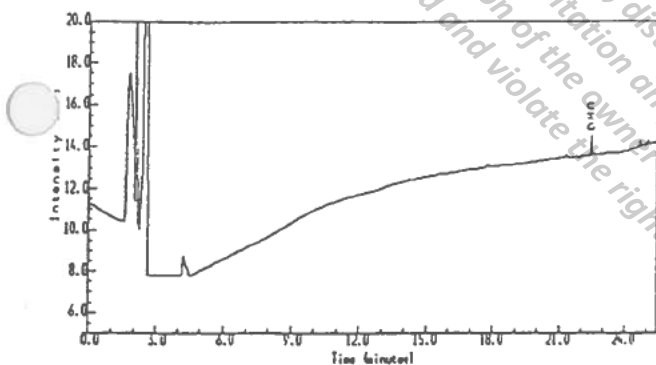
C-G Multichrom 1.8 (BONES)

1

2

Acquired on 9-SEP-1991 at 15:12

Acquired on 9-SEP-1991 at 16:31



C-G Multichrom 1.8 (BONES)

C-G Multichrom 1.8 (BONES)

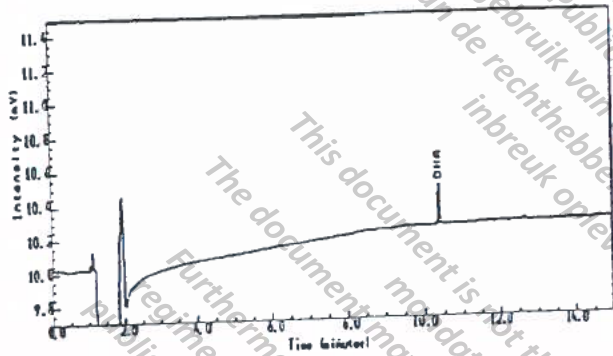
3

4

1. standard, DMA, 0.20 ng
2. 1-1-A, control, 1.829 mg injected, 0.039 ng found, 0.06 ppm
3. 1-1-A + 1.0 ppm, 0.457 mg injected, 0.197 ng found, 0.995 ppm found, 0.946 ppm corrected for control, 95% recovery
4. 2-1-A, treated sample, 0.915 mg injected, 0.176 ng found, 0.57 ppm

FIGURE 4. REPRESENTATIVE CHROMATOGRAMS FROM THE ANALYSIS OF POTATOES
(Storage Interval 0 Days)

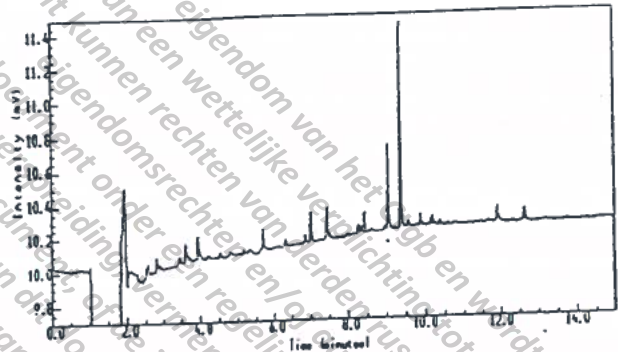
Acquired on 11-APR-1990 at 16:50



C-G Multichrom 1.6b(m)

3

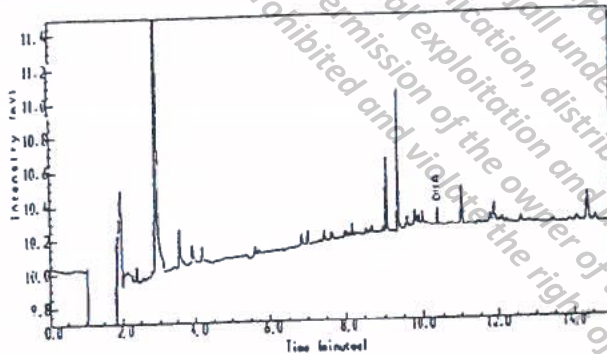
Acquired on 11-APR-1990 at 17:14



C-G Multichrom 1.6b(m)

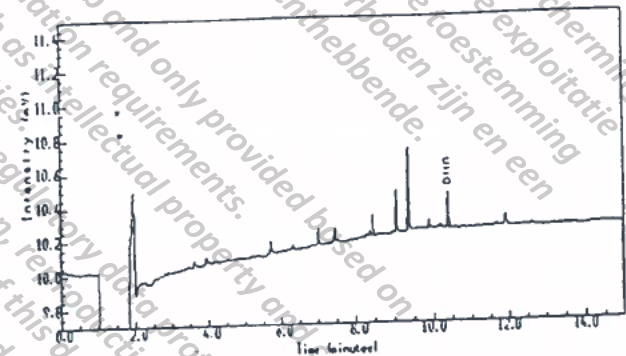
4

Acquired on 11-APR-1990 at 18:01



C-G Multichrom 1.6b(m)

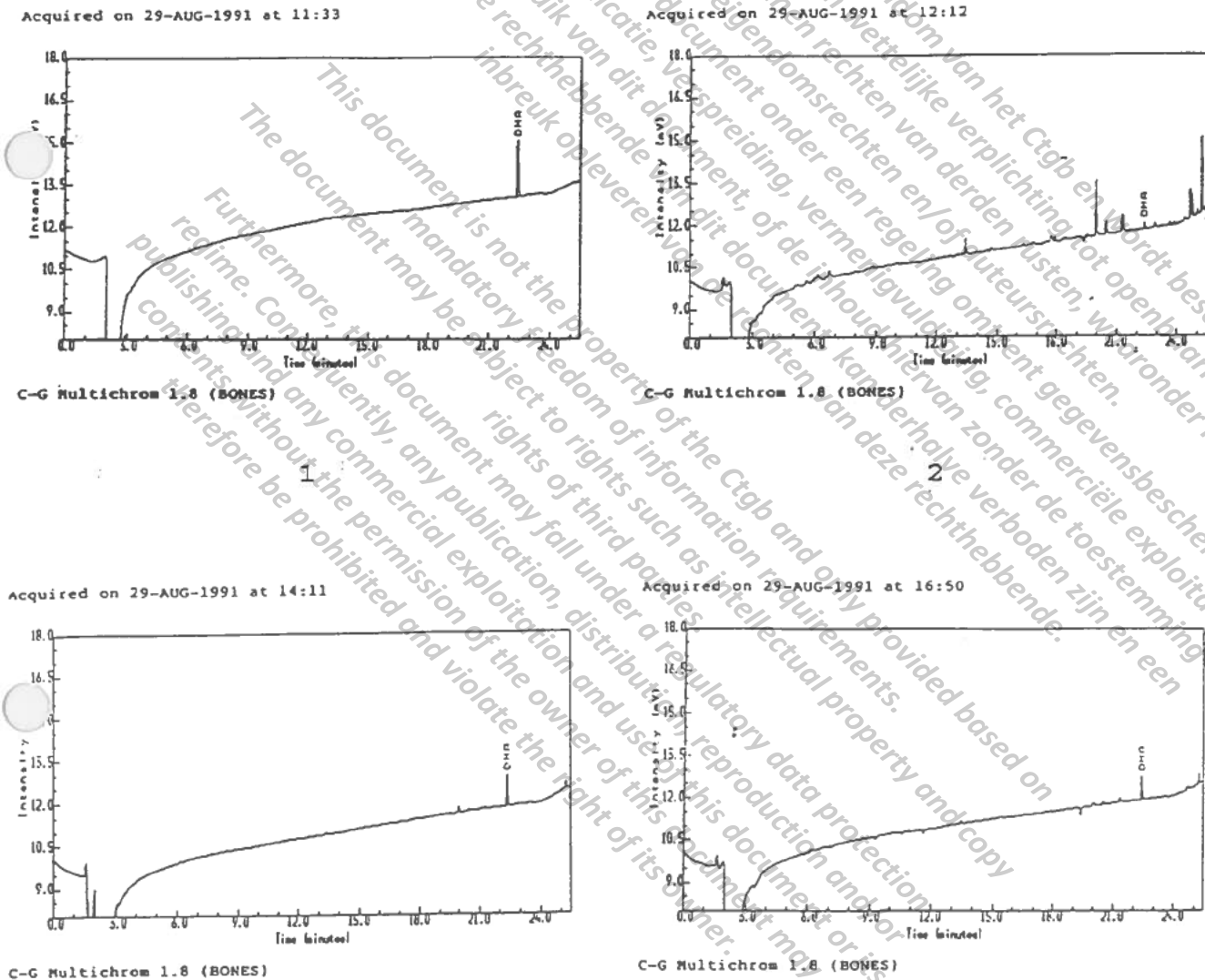
Acquired on 11-APR-1990 at 19:12



C-G Multichrom 1.6b(m)

1. standard, DMA, 0.10 ng
2. 1-1-A, control, 2.037 mg injected, (0.00 ng) <0.02 ng found, (0.00 ppm) <0.05 ppm
3. 1-1-A + 0.05 ppm, 2.037 mg injected, 0.043 ng found, 0.049 ppm found, 0.049 ppm corrected for control, 98% recovery
4. 4-1-A, treated sample, 1.019 mg injected, 0.090 ng found, 0.22 ppm

FIGURE 5. REPRESENTATIVE CHROMATOGRAMS FROM THE ANALYSIS OF SPINACH (Storage Interval 568 Days)

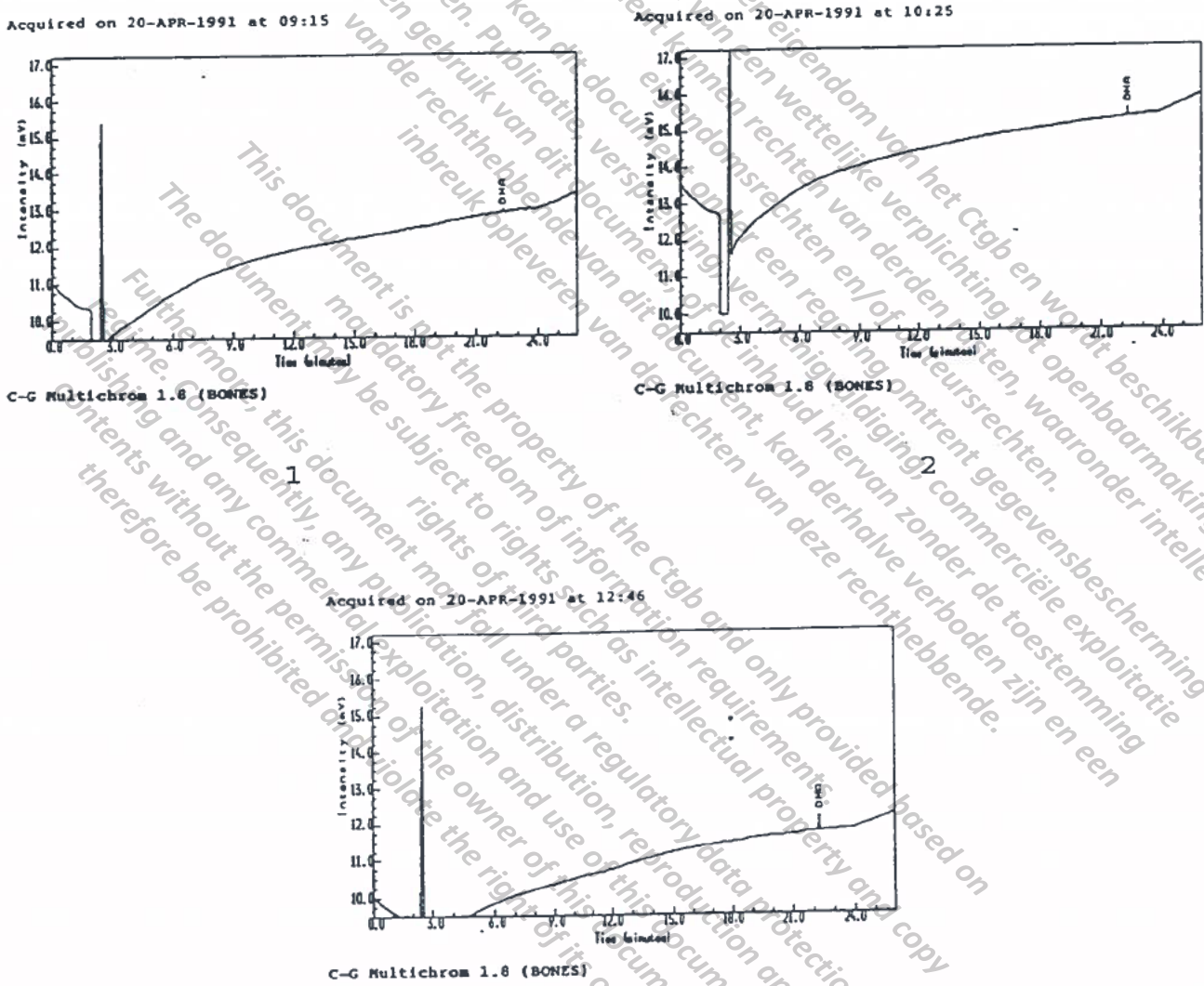


3

4

1. standard, DMA, 0.40 ng
2. 1-3-A, control, 1.834 mg injected, 0.053 ng found, 0.09 ppm
3. 1-3-A + 3.0 ppm, 0.183 mg injected, 0.222 ng found, 2.799 ppm found, 2.733 ppm corrected for control, 91% recovery
4. 2-3-A, treated sample, 0.244 mg injected, 0.173 ng found, 2.1 ppm

FIGURE 6. REPRESENTATIVE CHROMATOGRAMS OF DMA STANDARDS
(Cranberries, Storage Interval 186 Days)

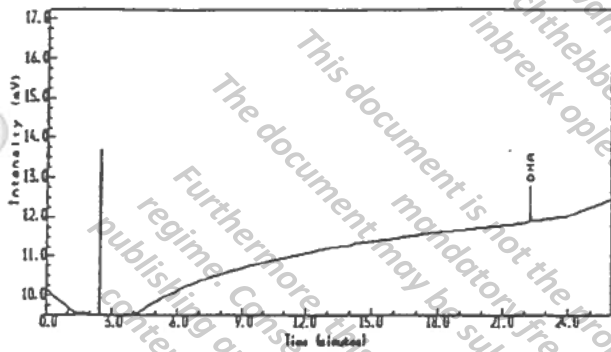


3

1. standard, DMA, 0.02 ng (detector response = 68)
2. standard, DMA, 0.05 ng (detector response = 216)
3. standard, DMA, 0.10 ng (detector response = 426)

FIGURE 6. REPRESENTATIVE CHROMATOGRAMS OF DMA STANDARDS (continued)
(Cranberries, Storage Interval 186 Days)

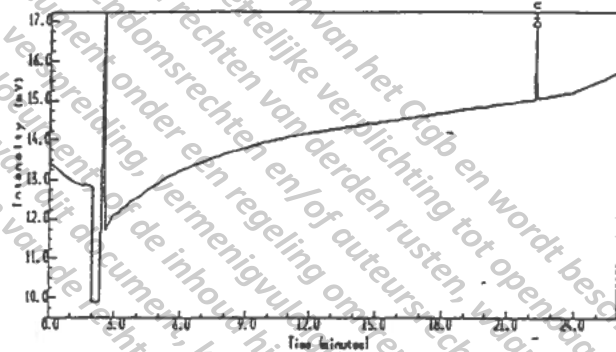
Acquired on 20-APR-1991 at 14:32



C-G Multichrom 1.8 (BONES)

4

Acquired on 20-APR-1991 at 11:36



C-G Multichrom 1.8 (BONES)

5

- 4. standard, DMA, 0.20 ng (detector response = 878)
- 5. standard, DMA, 0.40 ng (detector response = 1797)

FIGURE 7. REPRESENTATIVE CALIBRATION CURVE USING THE STANDARDS FROM FIGURE 6

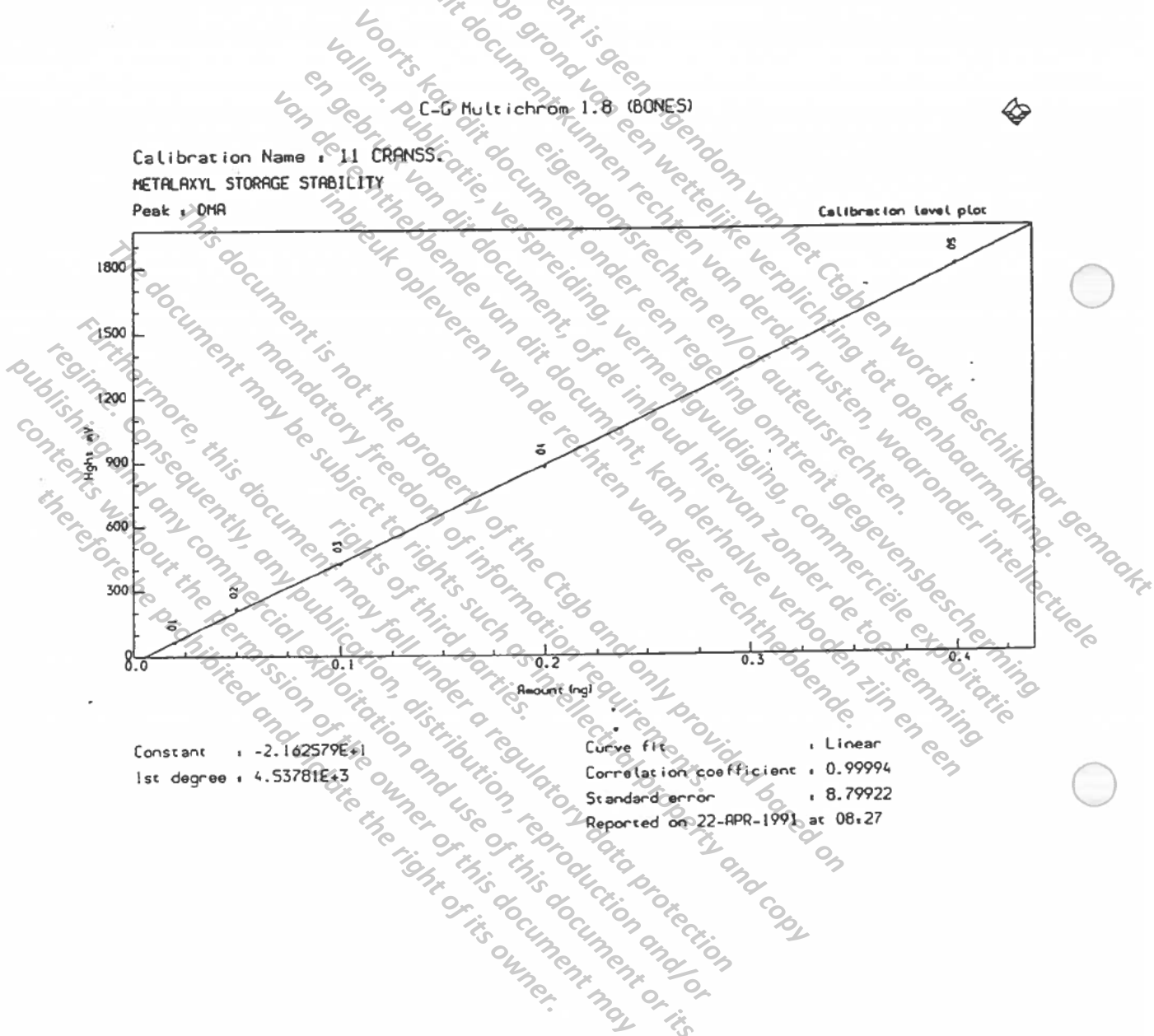


FIGURE 8. TOTAL RESIDUES OF METALAXYL DETERMINED
IN CRANBERRIES VERSUS FREEZER STORAGE
TIME

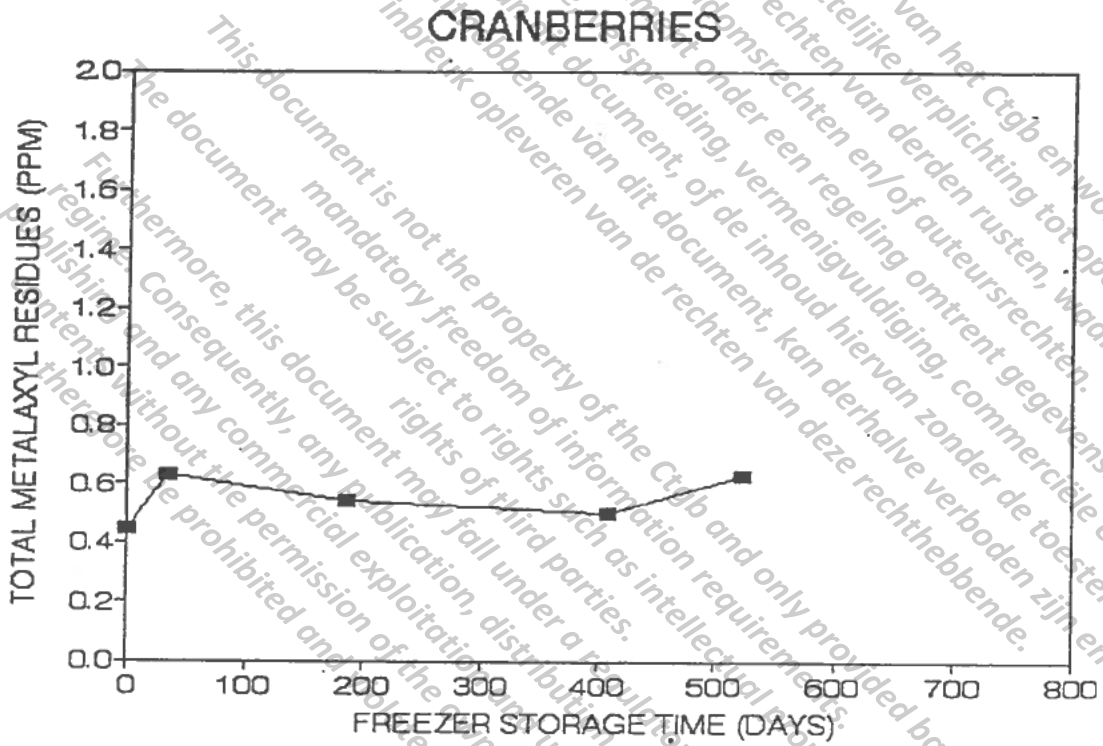


FIGURE 9. TOTAL RESIDUES OF METALAXYL DETERMINED
IN PEPPERS VERSUS FREEZER STORAGE TIME

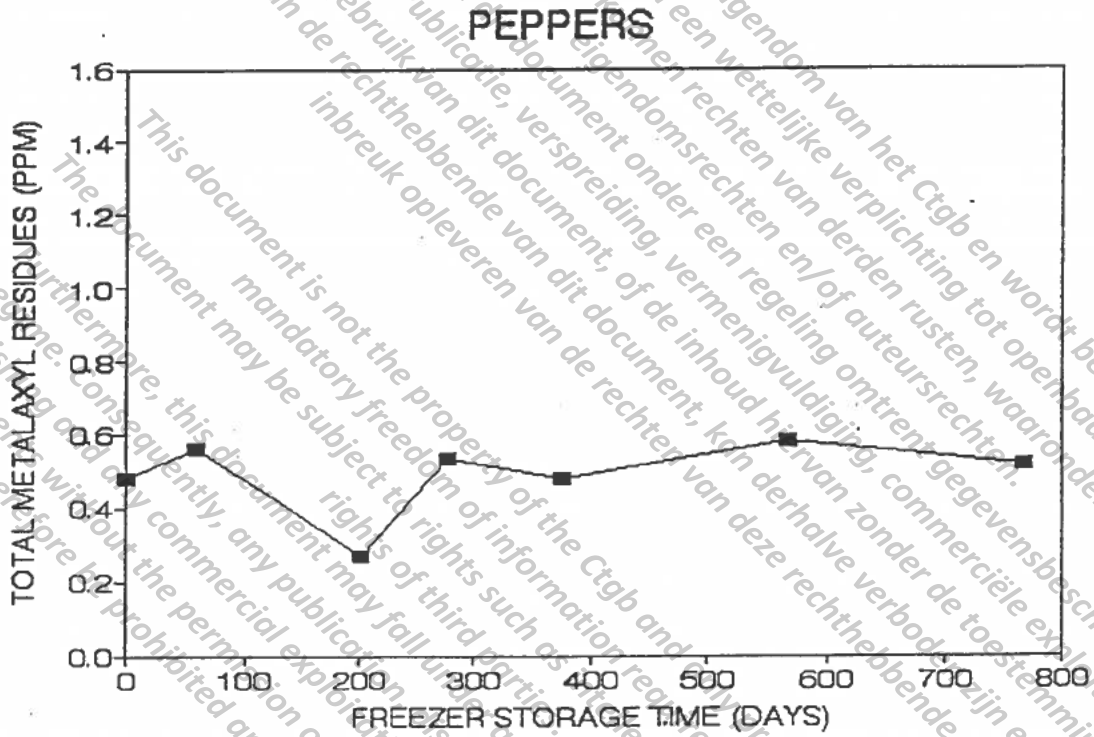


FIGURE 10. TOTAL RESIDUES OF METALAXYL DETERMINED IN POTATOES VERSUS FREEZER STORAGE TIME

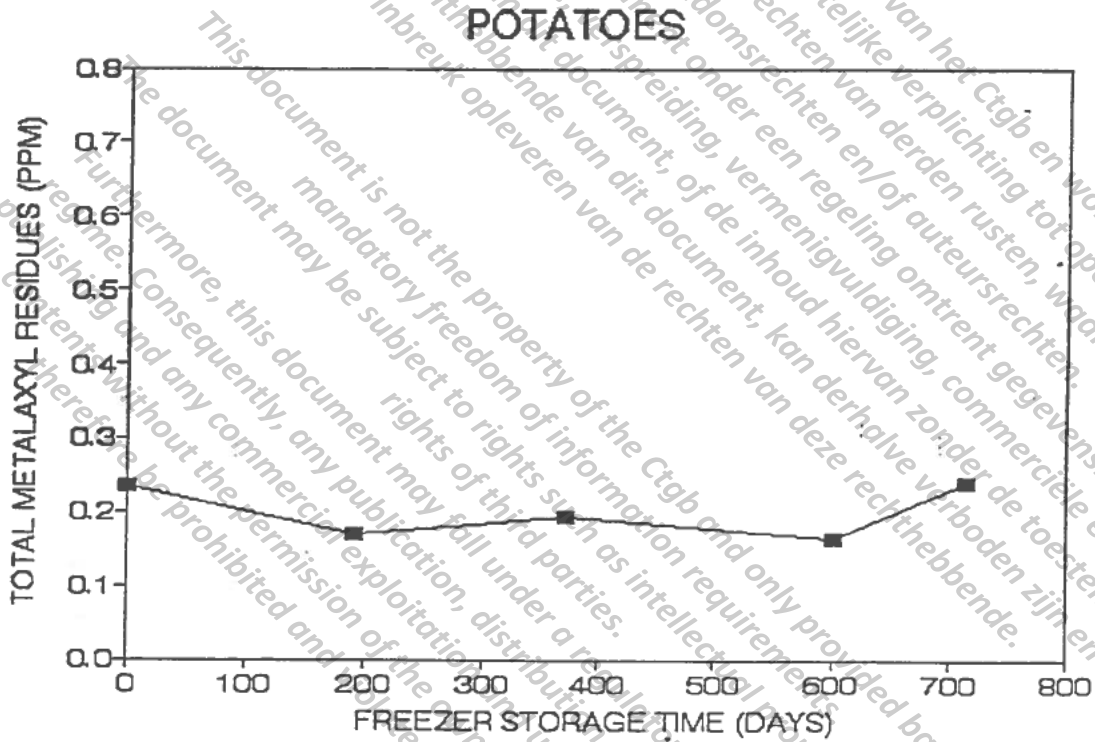
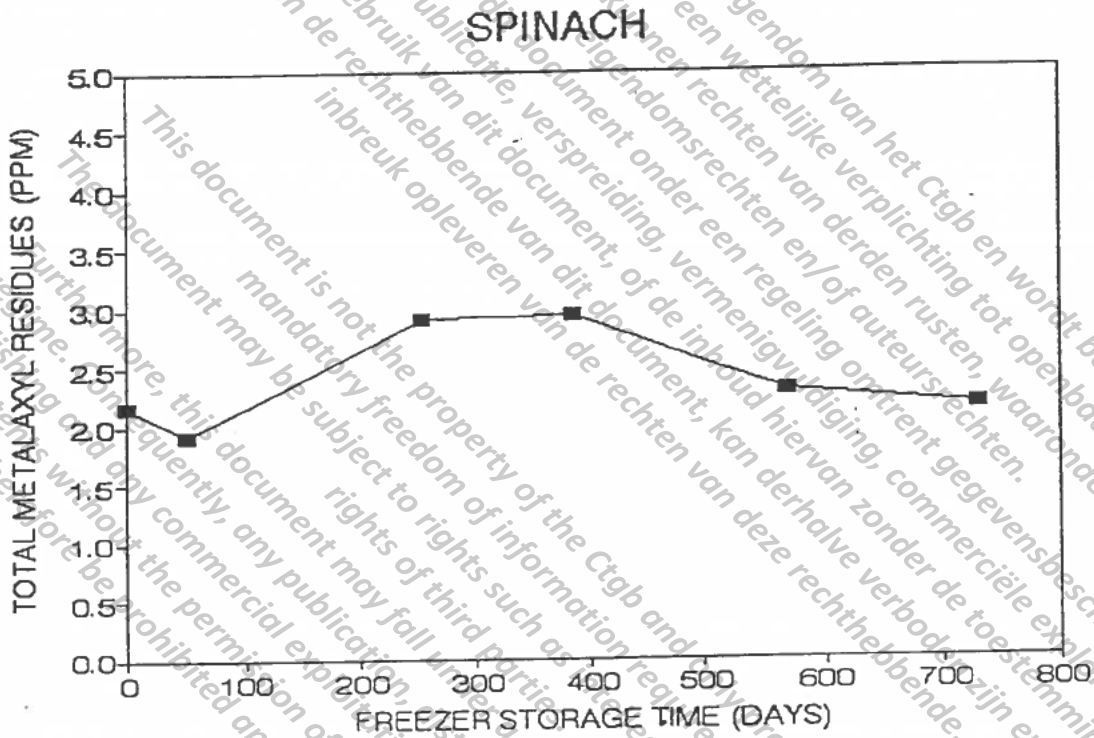


FIGURE 11. TOTAL RESIDUES OF METALAXYL DETERMINED
IN SPINACH VERSUS FREEZER STORAGE TIME



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5. Residue Test Report, RI-SS-012-90, Report 1
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