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I. SUMMARY AND INTRODUCTION

A. SCOPE

This method is for the confirmation of emamectin benzoate (MK-0244) and its isomer, 8,9-Z on representative samples of Crop Group 4: Leafy Vegetables and Crop Group 5: Brassica (Cole) Leafy Vegetables. Samples are prepared, extracted and analyzed by the normal procedures of Method 244-92-3, Revision 1. Method AG-684 allows for confirmation of residues by LC/MS.

B. PRINCIPLE

Samples are prepared, extracted, and analyzed using the procedures specified in analytical Method 244-92-3, Revision 1, "HPLC-Fluorescence Method to Determine the Total Toxic Residue of MK-0244 and its Metabolites on Vegetables, Including Leafy Vegetables and Cole Crops". Analysis of residues by this method utilizes derivatization with trifluoroacetic anhydride (TFAA) followed by HPLC with fluorescence detection. The confirmatory method described herein analyzes the underivatized fractions (found in Section IV. C. 18) by LC/MS.

II. MATERIALS AND METHODS

A. APPARATUS/EQUIPMENT

All apparatus are described in the analytical Method 244-92-3, Revision 1. Items considered to be equivalent to those listed in the method may be substituted after evaluation of the substituted item yields equivalent recoveries to those given in the method.

B. REAGENTS

Most reagents are described in the analytical Method 244-92-3, Revision 1. The additional reagents needed for the MS confirmation are:

27. LC/MS Standard solvent: 1% ammonium acetate in methanol (with 6% ethyl acetate). Dissolve 10.0 g of ammonium acetate in 1.0 L of methanol. Mix 940 mL of the 1% ammonium acetate in methanol with 60 mL of ethyl acetate.
28. LC/MS mobile phase A: acetonitrile
29. LC/MS mobile phase B: 0.1 M formic acid (Sigma cat. #F-0507, 99% pure or equivalent) in HPLC grade water (Mallinkrodt cat. # 6795 or equivalent). Dissolve 3.78 mL of formic acid in water and dilute to 1.0 L.

C. ANALYTICAL PROCEDURE

See Method 244-92-3, Revision 1, for the analytical procedures. Section IV C. 18 describes the splitting of the sample into two fractions. One fraction is derivatized and analyzed as described in subsequent method steps and the other is retained. Instead of retaining the second fraction for future use, as described in Method 244-92-3, Revision 1, it will be directly analyzed by LC/MS (and is not derivatized).

D. INSTRUMENTATION

1.0 Description and Operating Conditions

Residues are first determined using the instrumentation described in analytical Method 244-92-3, Revision 1. For confirmation of residues, the underivatized fraction found in Section IV C. 18 will be analyzed using the following parameters.

HPLC Conditions:

LC/MS Instrument: PE-Sciex API300 Mass Spectrometer with Atmospheric

Pressure Chemical Ionization  
(APCI) interface.  
HPLC Instrument: Shimadzu: SCL-10A controller,  
SIL-10-AXL injector, LC-10-AT  
pump.  
Eluent (Isocratic): 50/50 acetonitrile/0.1 M  
formic acid at 500 µL/min.  
Column: 3.0 x 250 mm Inertsil ODS-2,  
5µm Particle size,  
Temperature 35°C.  
Injection Volume: 15 µL.  
Retention Times: Emamectin Bla (~7 mins.),  
B1b (~6 mins.); Emamectin  
8,9-Z Bla (~9 mins.)  
Run Time: 10 minutes.  
Standard/Sample Solvent: 1% ammonium acetate in  
methanol (w/6% ethyl  
acetate)

Mass Spectrometer Conditions:

Source: APCI with Needle Current  
of +3uA, Temperature at  
375°C and Nitrogen as  
nebulizer gas.  
MS Mode: Single Quad Positive SIM  
MS Parameters: Orifice/Ring = 50/300  
Mass Calibration: Based on PPG masses: 59,  
175.133, 616.464,  
906.673, 1254.925,  
1545.134, 2010.469,  
2242.637  
Masses Monitored/Dwell: 872.7/1200ms(B1b),  
886.7/1200ms(B1a and  
8,9-Z).

2.0 Standardization: LC/MS

Calibrate and tune the mass spectrometer  
prior to analyzing samples. Check the  
calibration and tune by infusing a  
standard solution of polypropylene glycol  
(PPG) into the mass spectrometer using the  
IonSpray interface while monitoring

positive ions. Typical mass calibration tune files with PPG are presented in Figure 24. Monthly calibrations and tunes with the PPG solution are considered sufficient provided that instrument mass calibration stability is demonstrated for that time interval.

Determine the specific ion to monitor for each analyte by infusion of an analyte test solution (approx. 100 ng/ $\mu$ L in 50/50 acetonitrile/0.1 M formic acid) at 5  $\mu$ L/min. while scanning the Q1 quadrupole mass analyzer to find the optimum ion (See Figure 23).

E. INTERFERENCES

No interferences have been found by fluorescence detection or by LC/MS.

F. TIME REQUIRED

Approximately one day is required by an experienced analyst to analyze a set of 8 samples (including controls and procedural recoveries). Additional time is required to annotate and calculate the data.

G. PREPARATION OF STANDARD SOLUTIONS

The quantitation standard solution preparations for fluorescence analyses are specified in Method 244-92-3, Revision 1. Only MK-0244 and its 8,9-Z isomer are analyzed (See Figure 1). For the confirmatory analysis, the standards are not derivatized. The LC/MS quantitation standards for emamectin B1a and the 8,9-Z isomer are prepared as combined standards and contain equal concentrations of both compounds ranging from 1.0 to 10 ppb. The emamectin B1b quantitation standards are prepared separately

(from the B1a and 8,9-Z) and the concentration range is 0.523 to 5.23 ppb.

H. METHODS OF CALCULATION

All raw data for fluorescence analyses were processed using a Hewlett Packard HP3396A integrator. All raw data for LC/MS analyses were processed using PE Sciex Masschrom 1.0 instrument software. Calculations were performed, for both fluorescence and MS analyses in the same manner, using Quattro Pro software, incorporating the following formulas:

1. Calculation of Aliquot gram-Equivalent

$$\text{Aliquot g-equiv.} = \frac{\text{g sample extracted} \times \text{mL sample aliquot}}{\text{mL extraction volume}}$$

2. Calculation of mg-Equivalent Injected

$$\text{mg-equiv. injected} = \frac{\text{aliquot g-equiv.} \times (1000 \text{ mg/g}) \times \mu\text{L injected}}{\mu\text{L final volume} \times \text{dilution factor}}$$

3. Calculation of pg Found

$$\text{pg found} = \frac{\text{peak area (counts)} - \text{standard curve y-intercept (counts)}}{\text{standard curve slope (counts/pg)}}$$

D. Calculation of ppb Found

$$\text{ppb found} = \frac{\text{pg found}}{\text{mg-equivalent injected}}$$

E. Calculation of Percent Recovery

$$\% \text{ Recovery} = \frac{\text{ppb sample found}}{\text{fortification level (ppb)}} \times 100$$

F. Example Calculations for Broccoli Procedural Recovery Samples by LC/MS

Broccoli Procedural Recovery Sample for Bla, EP4797-S7,  
 Set # 2-01-MV, HPLC Run #68166, see Figure # 43

Sample wt.	= 10.0 g
Fortification Level	= 50 ppb
Extraction volume	= 40.0 mL
Aliquot volume	= 40.0 mL
Final volume	= 5.5 mL (5,500 µL)
Dilution factor	= 10
Injection volume	= 15.0 µL
Peak area (sample)	= 6825 counts
y-intercept	= -265.93 counts
Slope	= 130.18 counts/ng

$$\text{g-equiv.} = \frac{10.0 \text{ g} \times 40.0 \text{ mL}}{40.0 \text{ mL}} = 10.0 \text{ g}$$

$$\text{mg-equiv. injected} = \frac{10.0 \text{ g} \times (1000 \text{ mg/g}) \times 15 \text{ µL}}{11,000 \text{ µL} \times 10} = 1.364 \text{ mg}$$

$$\text{pg found} = \frac{6825 \text{ counts} - (-265.93) \text{ counts}}{130.18 \text{ counts/pg}} = 54.47 \text{ pg}$$

$$\text{ppb found} = \frac{54.47 \text{ pg}}{1.364 \text{ mg}} = 39.93 \text{ ppb}$$

$$\% \text{ recovery} = \frac{39.93 \text{ ppb}}{50 \text{ ppb}} \times 100 = 80 \%$$

The methods of calculation for fluorescence analysis are specified in Method 244-92-3, Revision 1.

### III. RESULTS AND DISCUSSION

All samples, control matrix and fresh laboratory fortifications of control matrix, were analyzed using method 244-92-3 with additional confirmation analyses as described in this (AG-684) document.

Emamectin Bla standard curves were used to quantitate emamectin Bla, emamectin Blb, and the 8,9-Z isomer using fluorescence detection (Figures 2-6). Figure 7 is the corresponding calibration curve using data from Figures 2-6. Figures 8-22 show representative chromatography of a reagent blank, a control for each matrix, and control matrices fortified at LOQ (5 ppb), at a mid-range level (10 or 15 ppb), and at 10x LOQ (50 ppb) for each matrix using fluorescence detection. Included in these are chromatograms showing quantitation of emamectin Blb recoveries (Figures 13 and 18), fortified at 2.62 ppb, from the 50 ppb Bla fortifications (the Bla standard contains approximately 5.23 % Blb).

Figures 23 and 24 illustrate the mass spectral scan of infused standards and a typical mass calibration for the LC/MS analyses.

Quantitation by LC/MS requires a standard curve be generated for each analyte. Figures 25-29 show typical emamectin Bla and 8,9-Z isomer standard chromatograms (from combined solutions; both compounds are quantitated in the same run). Figures 30-34 illustrate typical emamectin Blb standard chromatograms (quantitated in a separate run). Graphs for each of these sets of standards can be found in Figures 35-37. Figures 38-55 show representative chromatography of a reagent blank, a control for each matrix, and control matrices fortified at LOQ (5 ppb), at a mid-range level (10 or 15 ppb), and at 10x LOQ (50 ppb) for each matrix using MS detection. Included in these are

chromatograms showing quantitation of emamectin B1b recoveries (Figures 44 and 51), fortified at 2.62 ppb, from the 50 ppb B1a fortifications (the B1a standard contains 5.23 % B1b).

Recoveries for broccoli, fortified at 5, 10 and 50 ppb B1a and 8,9-Z (and 2.62 ppb B1b), were 87%  $\pm$  9% (n=7) by fluorescence and 86%  $\pm$  13% (n=7) by MS. Recoveries for celery, fortified at 5, 15, and 50 ppb B1a and 8,9-Z (and 2.62 ppb B1b), were 101%  $\pm$  7% (n=7) by fluorescence and 86%  $\pm$  5% (n=7) by MS. Recoveries for lettuce, fortified at 5, 10 and 50 ppb B1a and 8,9-Z (and 2.62 ppb B1b), were 109%  $\pm$  17% (n=7) by fluorescence and 95%  $\pm$  12% (n=7) by MS. Recoveries for each sample and each matrix are presented in Tables I to III.

#### IV. CONCLUSIONS

Method AG-684 is a valid and accurate method for the confirmation of MK-0244 and its 8,9-Z isomer in leafy vegetables and Brassica (cole) leafy vegetables using LC/MS. The limit of quantitation is 5 ppb.

V. CERTIFICATION

CERTIFICATION OF GOOD LABORATORY PRACTICES

This report and experimental results are certified to be authentic accounts of the procedure, observations, and data from Novartis Number 264-98.

Kent Ediger  
Kent Ediger  
Outside Research II

6/25/98  
Date

Table I  
 Emamectin Confirmatory Validation

Broccoli

EN-CAS ID #	Fortification Level (ppb)	Fort. Analyte	Set	Date Extracted	Fluorescence		LC/MS Date Injected, Bia and 8,9-Z	LC/MS Date Injected, Blb	Fluorescence Detection ppb Found <sup>1</sup> or % Recovery	LC/MS ppb Found or % Recovery (Bla)	LC/MS ppb Found or % Recovery (8,9-Z)	LC/MS ppb Found or % Recovery (Blb)
					Date Injected	Blb						
EP4797-C2	0		2-01-MV	4/14/98	4/15/98	4/17/98	4/17/98	4/17/98	<5	<5	<5	<5
EP4797-C3	0		2-02-MV	4/29/98	5/1/98	4/30/98	4/30/98	4/30/98	<5	<5	<5	<5
EP4797-C4	0		2-03-MV	5/11/98	5/12/98	5/11/98	5/11/98	5/11/98	<5 [ $<5$ ]	<5	<5	<5
EP4797-S13	5	MK-0244	2-03-MV	5/11/98	5/12/98	5/11/98	5/11/98	5/11/98	94%	111%	-	94%
EP4797-S14	5	8,9-Z	2-03-MV	5/11/98	5/12/98	5/11/98	5/11/98	5/11/98	94%	-	94%	-
EP4797-S11	10	MK-0244	2-02-MV	4/29/98	5/1/98	4/30/98	4/30/98	4/30/98	73%	85%	-	75%
EP4797-S12	10	8,9-Z	2-02-MV	4/29/98	5/1/98	4/30/98	4/30/98	4/30/98	74%	-	75%	-
EP4797-S7	50 [2.62] <sup>2</sup>	MK-0244	2-01-MV	4/14/98	4/15/98	4/17/98	4/17/98	4/17/98	92% [93%]	80%	-	85%
EP4797-S8	50	8,9-Z	2-01-MV	4/14/98	4/15/98	4/17/98	4/17/98	4/17/98	89%	-	74%	-
Reag. Blk.	0		2-01-MV	4/14/98	4/15/98	4/17/98	4/17/98	4/17/98	<5	<5	<5	<5

Mean and Std. Dev.:  
 By fluorescence = 87% ± 9% (n=7)  
 By LC/MS = 86% ± 13% (n=7)

<sup>1</sup> Blb values are bracketed.  
<sup>2</sup> Blb fortification of 50 ppb corresponds to 2.62 ppb Blb

Table II  
 Emamectin Confirmatory Validation

Celery

EN-CAS ID #	Fortification Level (ppb)	Fort. Analyte	Set	Date Extracted	Fluorescence		LC/MS Date Injected, Bla and 8,9-Z	LC/MS Date Injected, Blb	Fluorescence Detection ppb Found <sup>1</sup> or % Recovery	LC/MS ppb Found or % Recovery (Bla)	LC/MS ppb Found or % Recovery (8,9-Z)	LC/MS ppb Found or % Recovery (Blb)
					Detection Date Injected	Detection						
EP4798-C2	0		3-01-MV	4/20/98	4/22/98	4/22/98	4/22/98	4/30/98	<5	<5	<5	<5
EP4798-C3	0		3-02-MV	4/29/98	5/1/98 <sup>3</sup>	4/30/98	4/30/98	4/30/98	<5 [ $<5$ ]	<5	-	-
EP4798-S3	5	MK-0244	3-01-MV	4/20/98	4/22/98	4/22/98	4/22/98	4/22/98	105%	90%	-	-
EP4798-S4	5	8,9-Z	3-01-MV	4/20/98	4/22/98	4/22/98	4/22/98	4/22/98	105%	-	83%	-
EP4798-S9	15	MK-0244	3-02-MV	4/29/98	5/1/98	4/30/98	4/30/98	4/30/98	95%	78%	-	-
EP4798-S6	15	8,9-Z	3-01-MV	4/20/98	4/22/98	4/22/98	4/22/98	4/22/98	100%	-	87%	-
EP4798-S10	50 [2.62] <sup>2</sup>	MK-0244	3-02-MV	4/29/98	5/1/98 <sup>3</sup>	4/30/98	4/30/98	4/30/98	105% [107%]	89%	-	93%
EP4798-S8	50	8,9-Z	3-01-MV	4/20/98	4/22/98	4/22/98	4/22/98	4/22/98	87%	-	82%	-
Reag. Blk.	0		3-01-MV	4/20/98	4/22/98	4/22/98	4/22/98	4/22/98	<5	<5	<5	<5

Mean and Std. Dev. :  
 By fluorescence = 101%  $\pm$  7% (n=7)  
 By LC/MS = 86%  $\pm$  5% (n=7)

<sup>1</sup> Blb values are bracketed.  
<sup>2</sup> Bla fortification of 50 ppb corresponds to 2.62 ppb Blb  
<sup>3</sup> Samples were re injected for Blb on 5/5/98

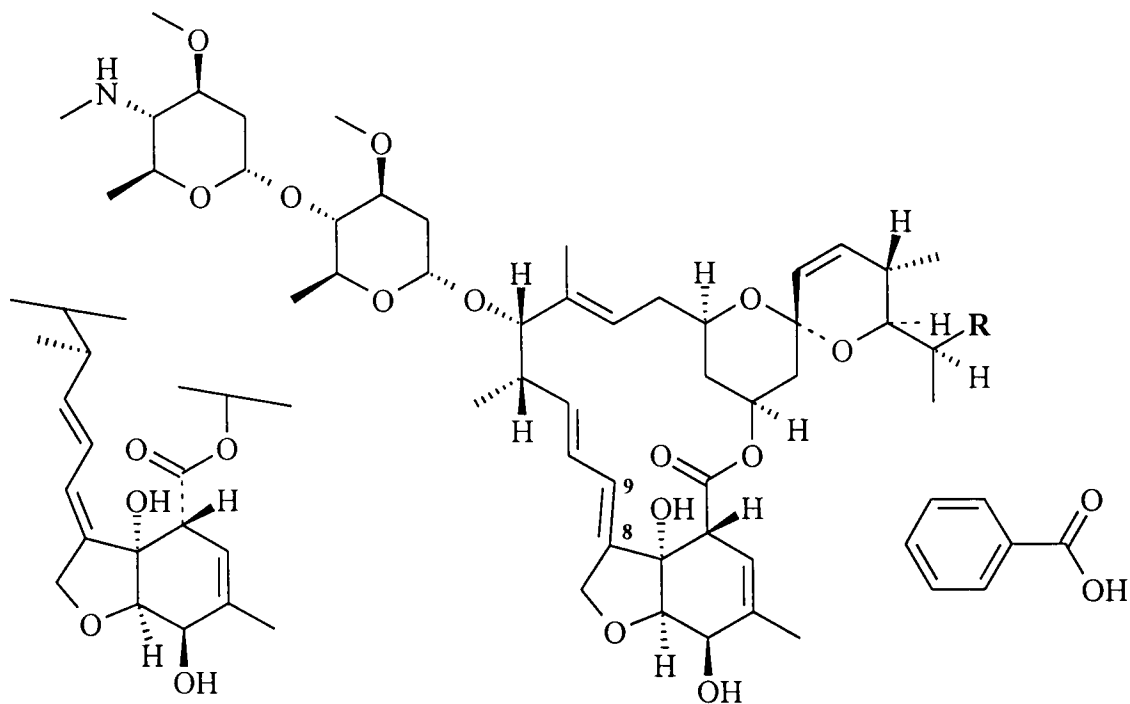
Table III  
 Emamectin Confirmatory Validation

EN-CAS ID #	Fortification Level (ppb)	Fort. Analyte	Set	Date Extracted	Fluorescence		LC/MS Date Injected, Bla and 8,9-Z	LC/MS Date Injected, Bla	Fluorescence Detection ppb Found <sup>1</sup> or % Recovery	LC/MS ppb Found or % Recovery (Bla)	LC/MS ppb Found or % Recovery (8,9-Z)	LC/MS ppb Found or % Recovery (Bib)
					Date Injected	Date						
EP4796-C2	0		1-01-MV	4/7/98	4/8/98	4/8/98	4/10/98	4/17/98	<5 [ $<5$ ]	<5	<5	<5
EP4796-S3	5	MK-0244	1-01-MV	4/7/98	4/8/98	4/8/98	4/10/98		111%	109%	-	
EP4796-S4	5	8,9-Z	1-01-MV	4/7/98	4/8/98	4/8/98	4/10/98		106%	-	96%	
EP4796-S5	10	MK-0244	1-01-MV	4/7/98	4/8/98	4/8/98	4/10/98		114%	108%	-	
EP4796-S6	10	8,9-Z	1-01-MV	4/7/98	4/8/98	4/8/98	4/10/98		74%	-	98%	89%
EP4796-S7	50[2.62] <sup>2</sup>	MK-0244	1-01-MV	4/7/98	4/8/98	4/8/98	4/10/98	4/17/98	112% [124%]	88%	-	
EP4796-S8	50	8,9-Z	1-01-MV	4/7/98	4/8/98	4/8/98	4/10/98		122%	-	75%	
Reag. Bik.	0		1-01-MV	4/7/98	4/8/98	4/8/98	4/10/98		<5	<5	<5	

Mean and Std. Dev. :  
 By fluorescence = 109% ± 17% (n=7)  
 By LC/MS = 95% ± 12% (n=7)

<sup>1</sup> Bib values are bracketed.  
<sup>2</sup> Bla fortification of 50 ppb corresponds to 2.62 ppb Bib

FIGURE 1  
Chemical Structures



8,9-Z Isomer

Emamectin B1 Benzoate Salt  
R=C<sub>2</sub>H<sub>5</sub> for Bla component  
R=CH<sub>3</sub> for B1b component

FIGURE 2  
Typical HPLC Fluorescence Detector Chromatogram  
1.0 ng/mL Standard, 50  $\mu$ L Injected, Set # 2-01-MV, Run # 69029

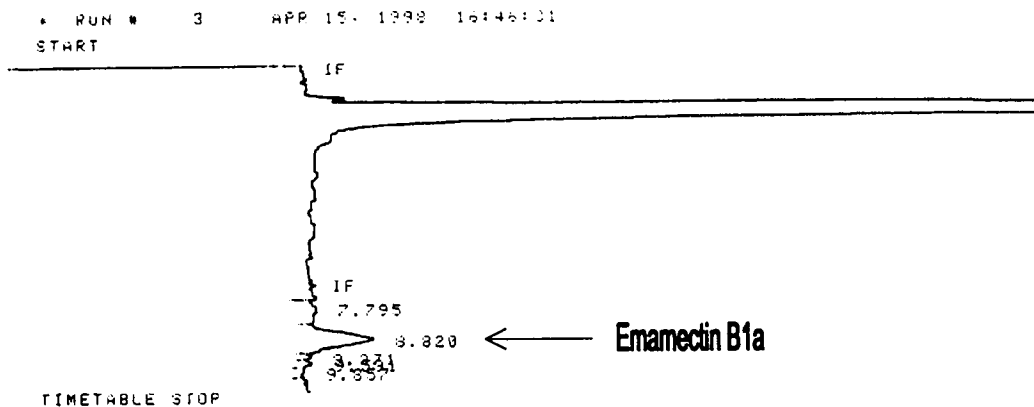


FIGURE 3  
Typical HPLC Fluorescence Detector Chromatogram  
2.0 ng/mL Standard, 50  $\mu$ L Injected, Set # 2-01-MV, Run # 69029

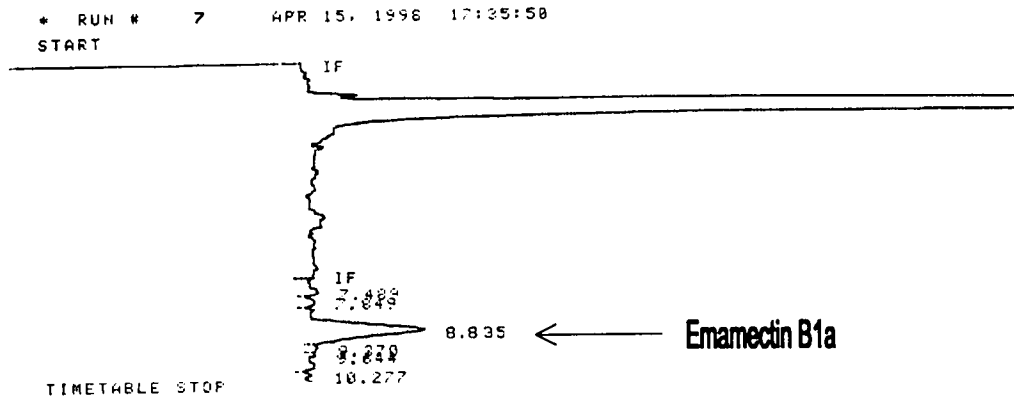


FIGURE 4  
Typical HPLC Fluorescence Detector Chromatogram  
5.0 ng/mL Standard, 50  $\mu$ L Injected, Set # 2-01-MV, Run # 69029

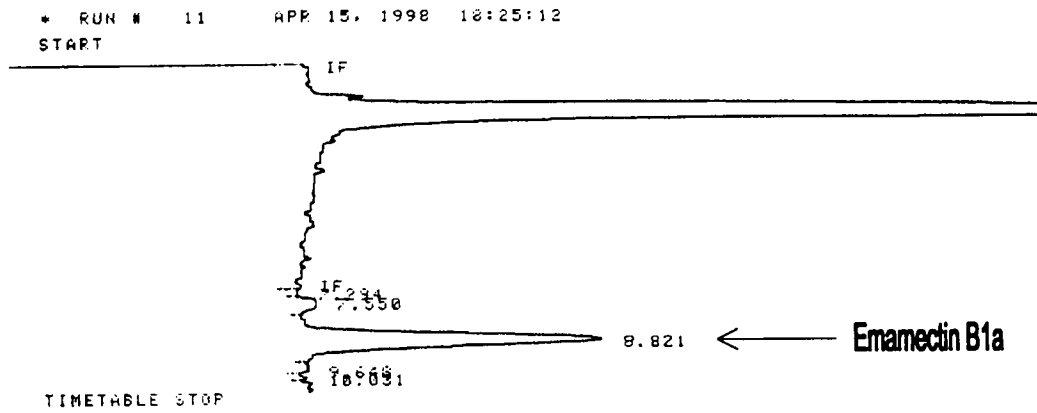


FIGURE 5  
Typical HPLC Fluorescence Detector Chromatogram  
8.0 ng/mL Standard, 50  $\mu$ L Injected, Set # 2-01-MV, Run # 69029

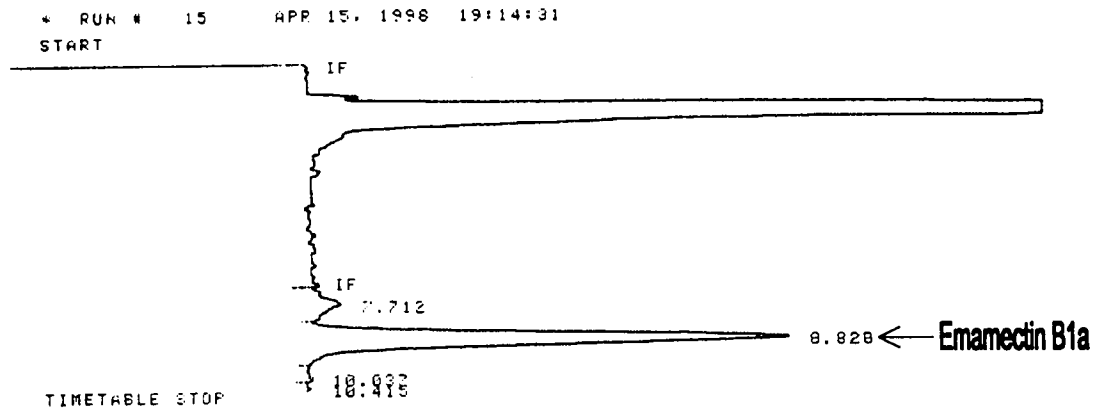


FIGURE 6  
Typical HPLC Fluorescence Detector Chromatogram  
10.0 ng/mL Standard, 50  $\mu$ L Injected, Set # 2-01-MV, Run # 69029

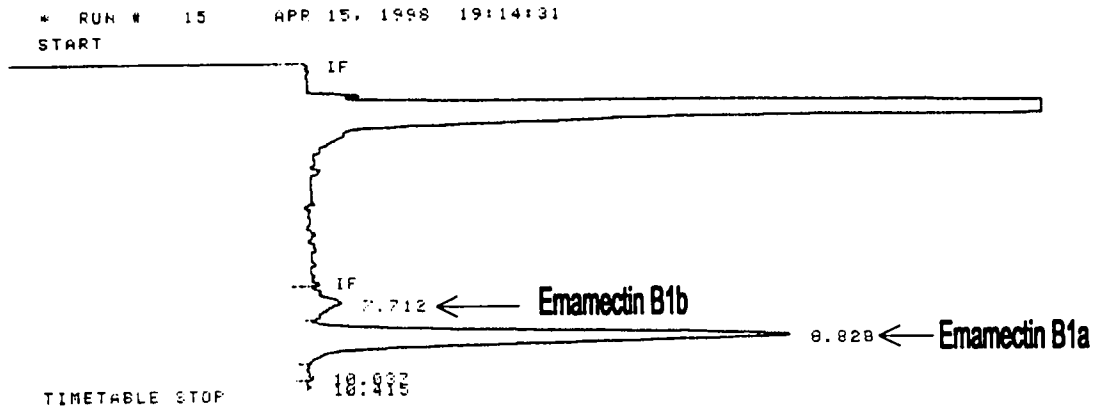


FIGURE 7  
 Typical HPLC/Fluorescence Detector Calibration Curve  
 Set # 2-01-MV, Run # 69029

LLS rev.2.0  
 CALC/GRAPH  
 EN-CAS LABS  
 04/10/92 dia

Proj.#: 98-0012  
 Set: 2-01-MV  
 Analyte: EMAMECTIN

CalcDate: 04/17/98  
 By(Init): ABR  
 RunNumbr 69029

*MP* 5-12-98

X	Y
50	141214
100	251397
250	616220
400	917531
500	1098818

STATISTICS		
Regression Output:		
Constant	45894.5	(y-int)
Std Err of Y Est	25421.23	
R Squared	0.997156	(variance)
No. of Observations	5	
Degrees of Freedom	3	
X Coefficient(s)	2150.544	(slope)
Std Err of Coef.	66.30372	
Corr. Coeff.	0.998577	

=====

98-0012 EMAMECTIN  
 2-01-MV BROCCOLI

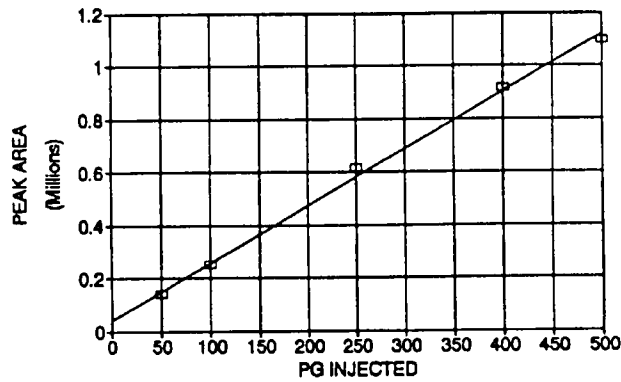


FIGURE 8  
Typical HPLC/Fluorescence Detector Chromatogram  
Reagent Blank, 50  $\mu$ L Injected, Set # 2-01-MV, Run # 69029  
EN-CAS Sample Code R. Blk

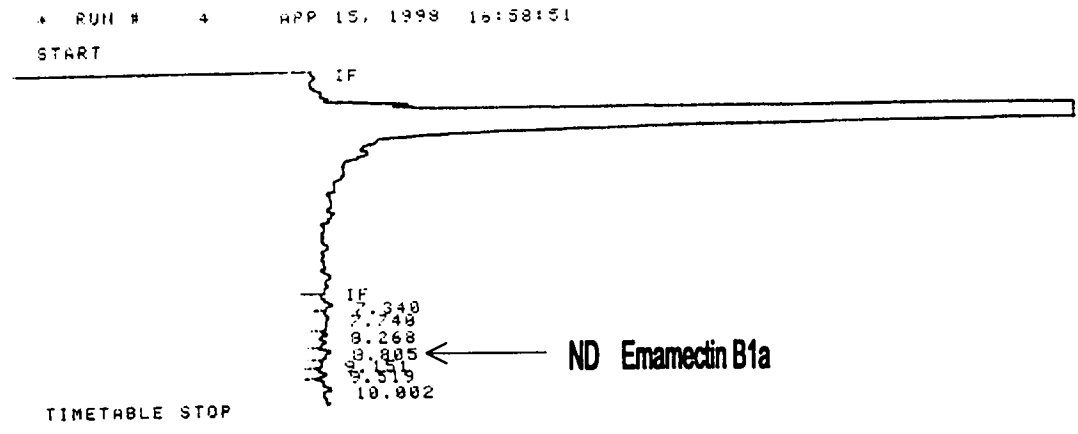


FIGURE 9  
Typical HPLC Fluorescence Detector Chromatogram  
Control Broccoli, 50  $\mu$ L Injected, Set # 2-01-MV, Run # 69029  
EN-CAS Sample Code EP4797-C2

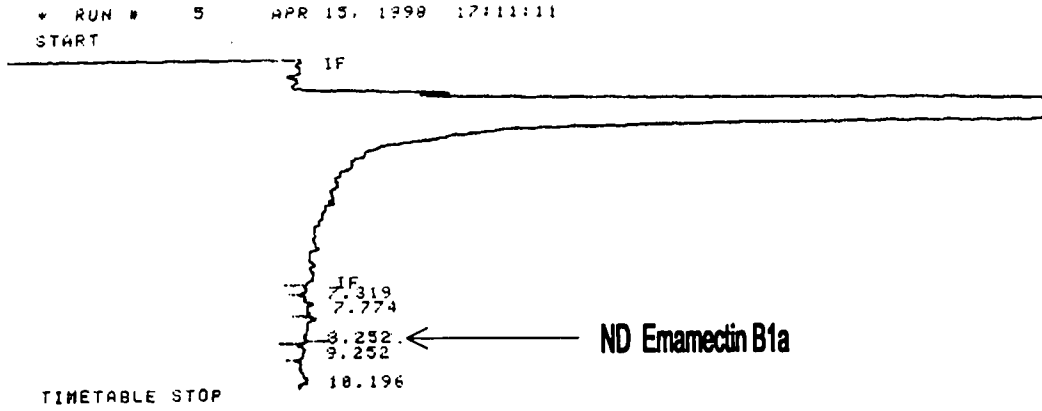


FIGURE 10  
Typical HPLC Fluorescence Detector Chromatogram  
Control Broccoli + 5 ppb MK-0244, 50  $\mu$ L Injected, Set # 2-03-MV, Run # 69116  
EN-CAS Sample Code EP4797-S13

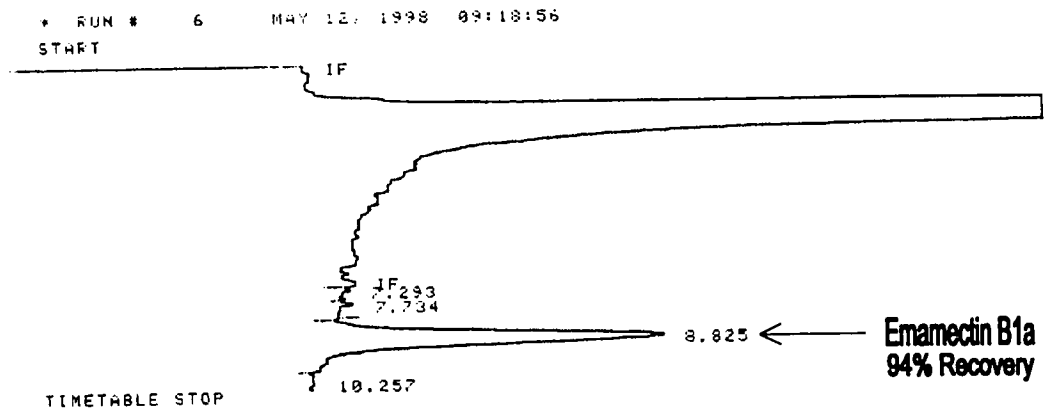


FIGURE 11  
Typical HPLC Fluorescence Detector Chromatogram  
Control Broccoli + 10 ppb MK-0244, 50  $\mu$ L Injected, Set # 2-02-MV, Run # 69080  
EN-CAS Sample Code EP4797-S11

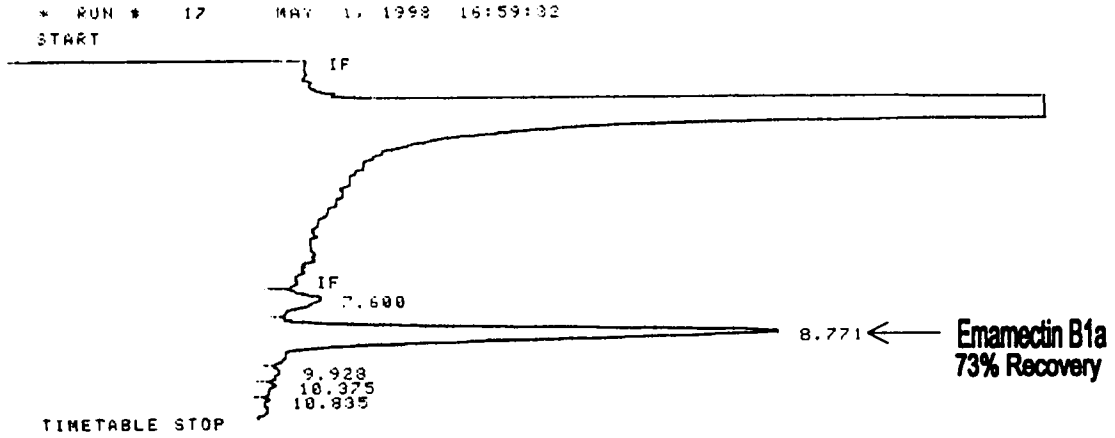


FIGURE 12  
Typical HPLC Fluorescence Detector Chromatogram  
Control Broccoli + 50 ppb MK-0244, 50  $\mu$ L Injected, Set # 2-01-MV, Run # 69029  
EN-CAS Sample Code EP4797-S7, 1 to 10 Dilution  
NOTE: See Figure 13 for undiluted injection for quantitation of Emamectin B1b.

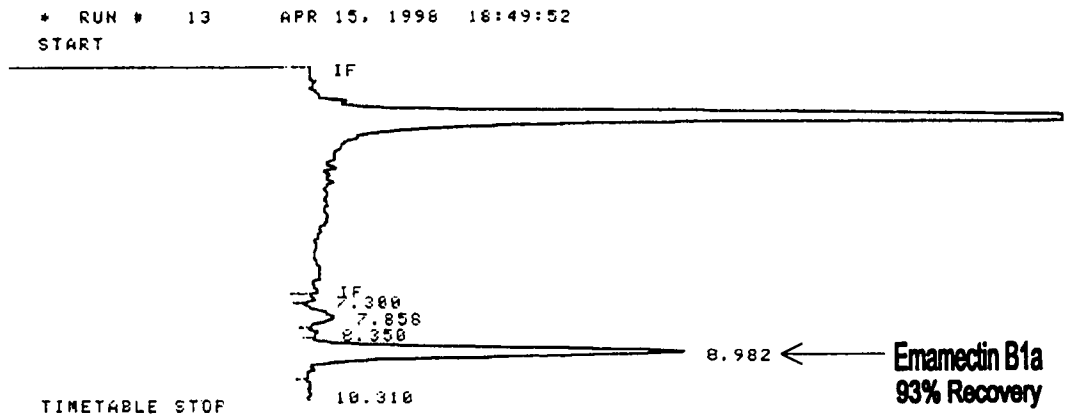


FIGURE 13  
Typical HPLC Fluorescence Detector Chromatogram  
Control Broccoli + 50 ppb MK-0244 (2.62 ppb B1b), 50  $\mu$ L Injected, Set # 2-01-MV,  
Run # 69029, EN-CAS Sample Code EP4797-S7  
NOTE: See Figure 12 for diluted injection for quantitation of Emamectin B1a.

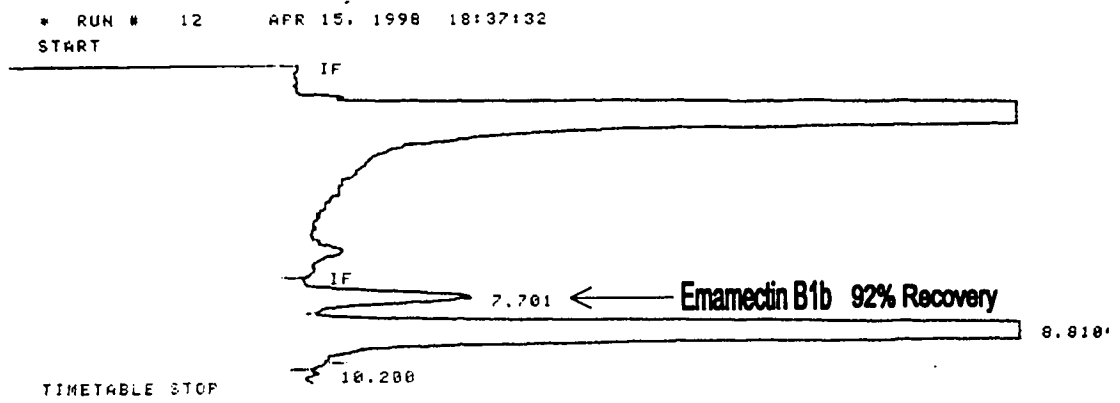


FIGURE 14  
Typical HPLC Fluorescence Detector Chromatogram  
Control Lettuce, 50  $\mu$ L Injected, Set # 1-01-MV, Run # 69012  
EN-CAS Sample Code EP4796-C2

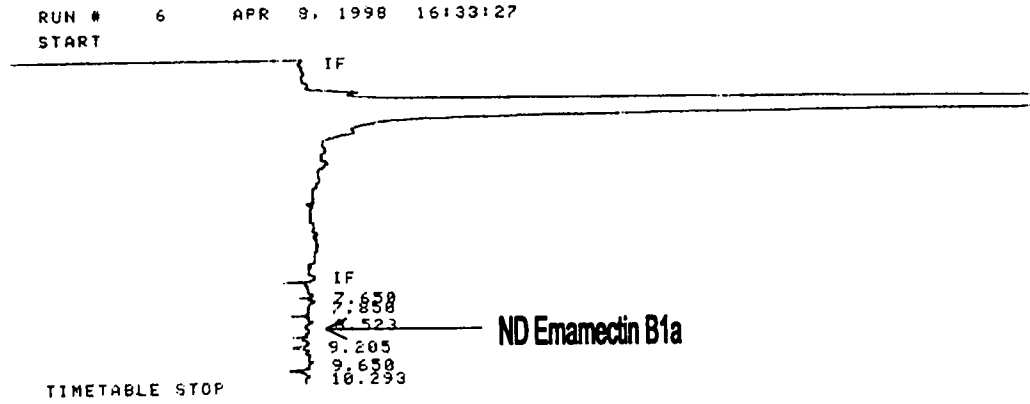


FIGURE 15  
Typical HPLC/Fluorescence Detector Chromatogram  
Control Lettuce + 5 ppb 8,9-Z, 50  $\mu$ L Injected, Set # 1-01-MV, Run # 69012  
EN-CAS Sample Code EP4796-S4

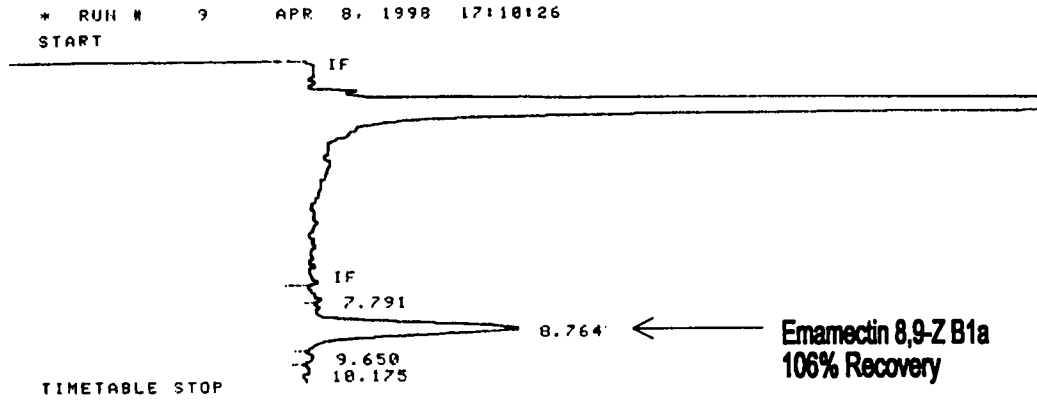


FIGURE 16  
Typical HPLC/Fluorescence Detector Chromatogram  
Control Lettuce + 10 ppb 8,9-Z, 50  $\mu$ L Injected, Set # 1-01-MV, Run # 69012  
EN-CAS Sample Code EP4796-S6

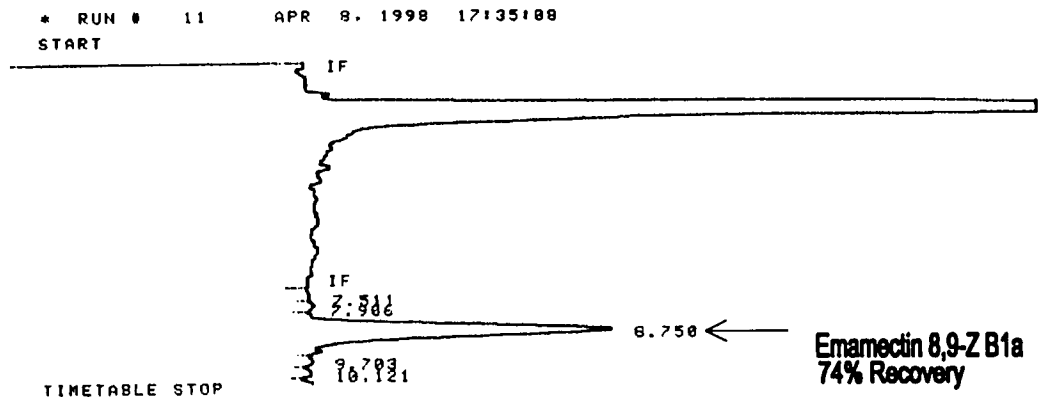
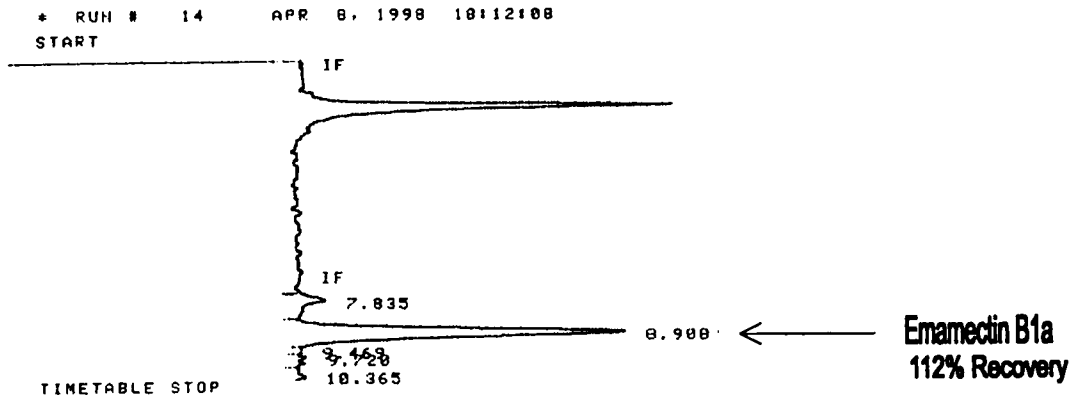


FIGURE 17  
Typical HPLC/Fluorescence Detector Chromatogram  
Control Lettuce + 50 ppb MK-0244, 50  $\mu$ L Injected, Set # 1-01-MV, Run # 69012  
EN-CAS Sample Code EP4796-S7, 1 to 10 Dilution  
NOTE: See Figure 18 for undiluted injection for quantitation of Emamectin B1b.



**FIGURE 18**  
Typical HPLC/Fluorescence Detector Chromatogram  
Control Lettuce + 50 ppb MK-0244 (2.62 ppb B1b), 50  $\mu$ L Injected, Set # 1-01-MV,  
Run # 69012, EN-CAS Sample Code EP4796-S7, 1 to 10 Dilution  
NOTE: See Figure 17 for diluted injection for quantitation of Emamectin B1a.

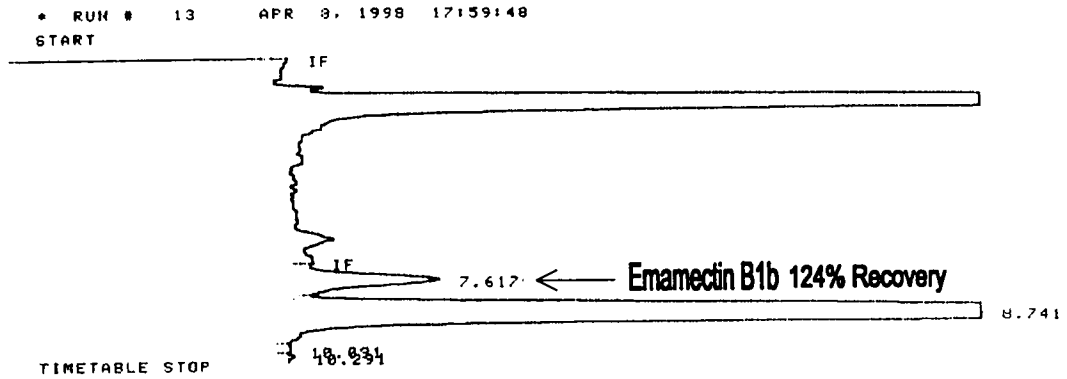


FIGURE 19  
Typical HPLC Fluorescence Detector Chromatogram  
Control Celery, 50  $\mu$ L Injected, Set # 3-01-MV, Run # 69048  
EN-CAS Sample Code EP4798-C2

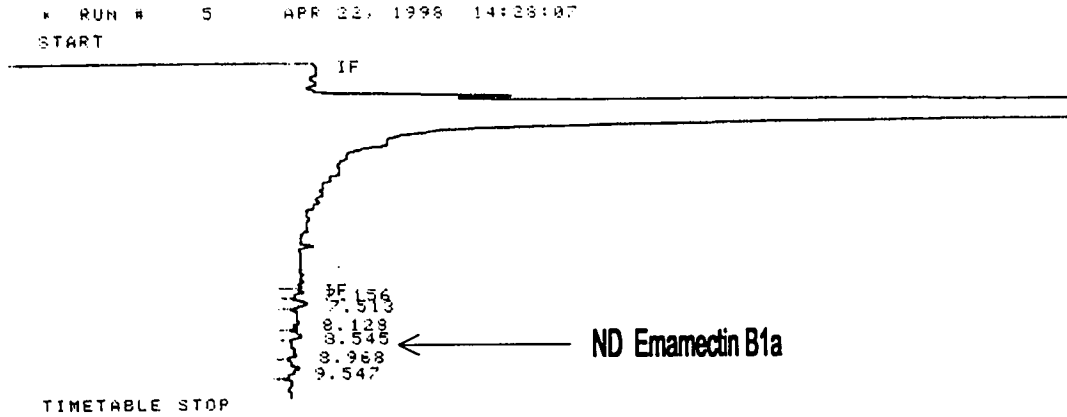


FIGURE 20  
Typical HPLC Fluorescence Detector Chromatogram  
Control Celery + 5 ppb Emamectin 8,9-Z, 50  $\mu$ L Injected, Set # 3-01-MV,  
Run # 69048, EN-CAS Sample Code EP4798-S4

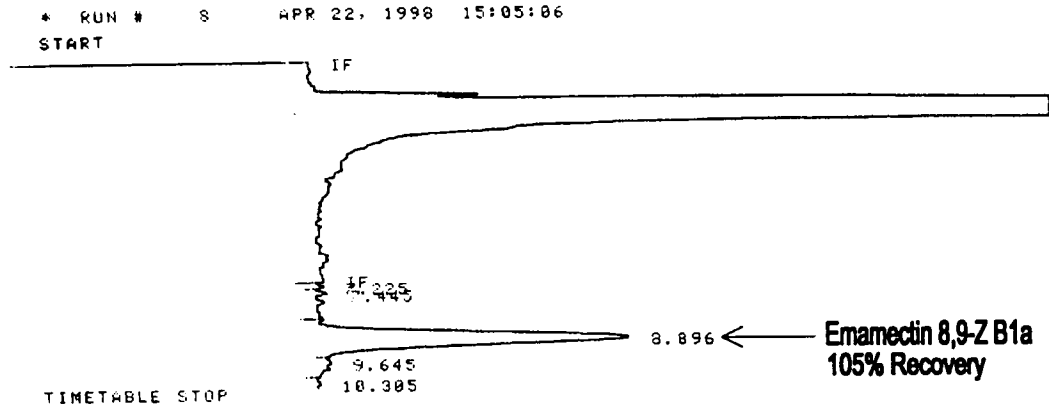


FIGURE 21  
Typical HPLC Fluorescence Detector Chromatogram  
Control Celery + 15 ppb Emamectin 8,9-Z, 50  $\mu$ L Injected, Set # 3-01-MV,  
Run # 69048, EN-CAS Sample Code EP4798-S6, 1 to 2 Dilution

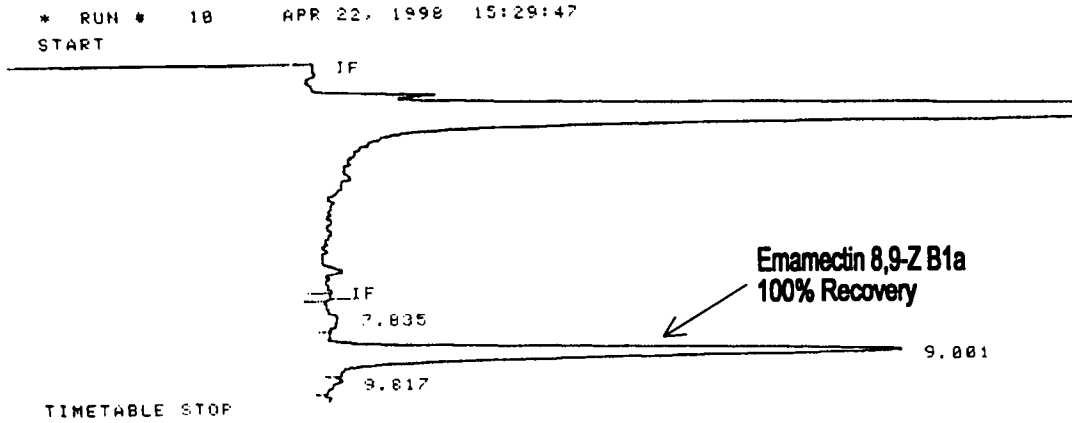


FIGURE 22  
Typical HPLC Fluorescence Detector Chromatogram  
Control Celery + 50 ppb Enamectin 8,9-Z, 50  $\mu$ L Injected, Set # 3-01-MV,  
Run # 69048, EN-CAS Sample Code EP4798-S8, 1 to 10 Dilution

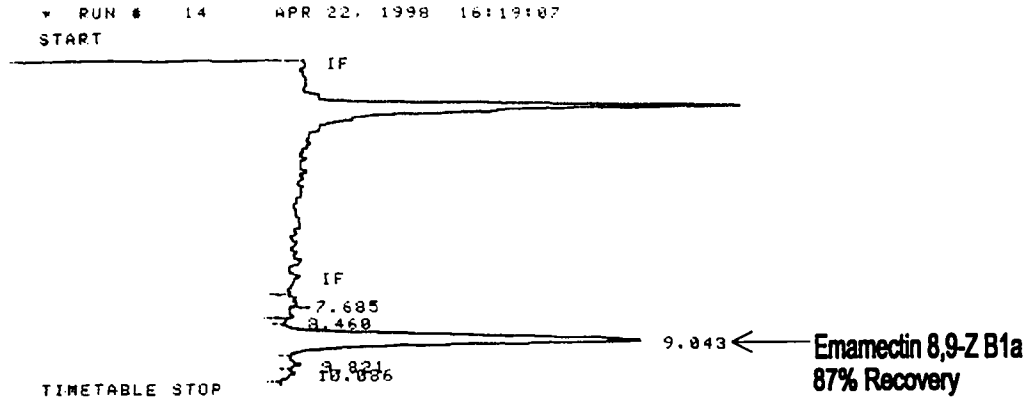


FIGURE 23  
LC/MS Scan of Infused Standards

MultiView 1.3 Friday, May 29, 1998 16:19 page 1 of 1  
Info for pane 1: emaB1#2 (\*\*Emamectin PIS (underiv)\*\*)  
Period 1, Expt. 1; Mass range: 110.0 to 1100.0 by 0.1 amu; Dwell: 10.0 ms; Pause: 5.0 ms  
Acq. Time: Wed, Nov 19, 1997 at 16:08:54; Exp. Comment: Emamectin PIS

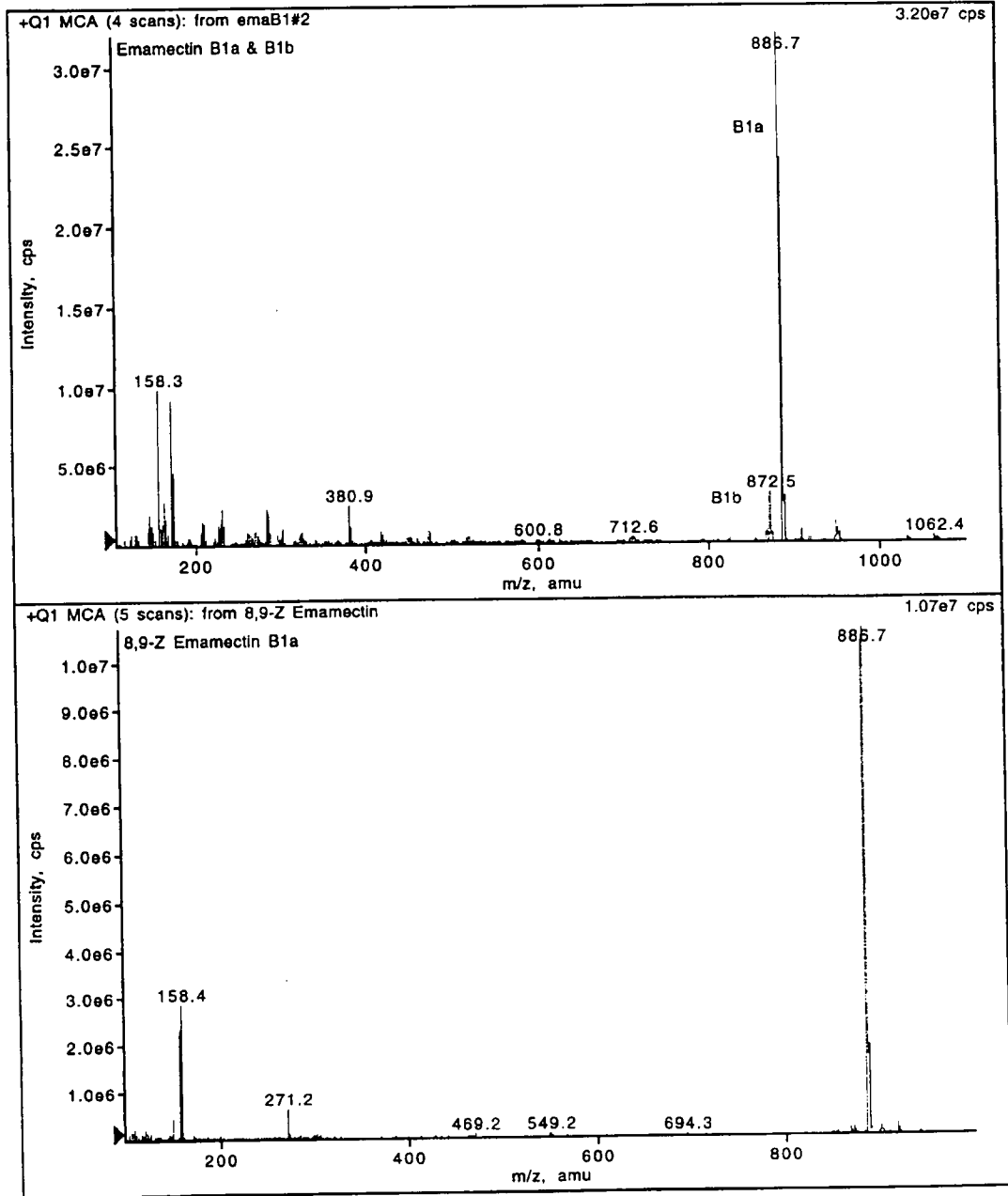


FIGURE 24  
 Mass Calibration Printout

TBB 4/14/98

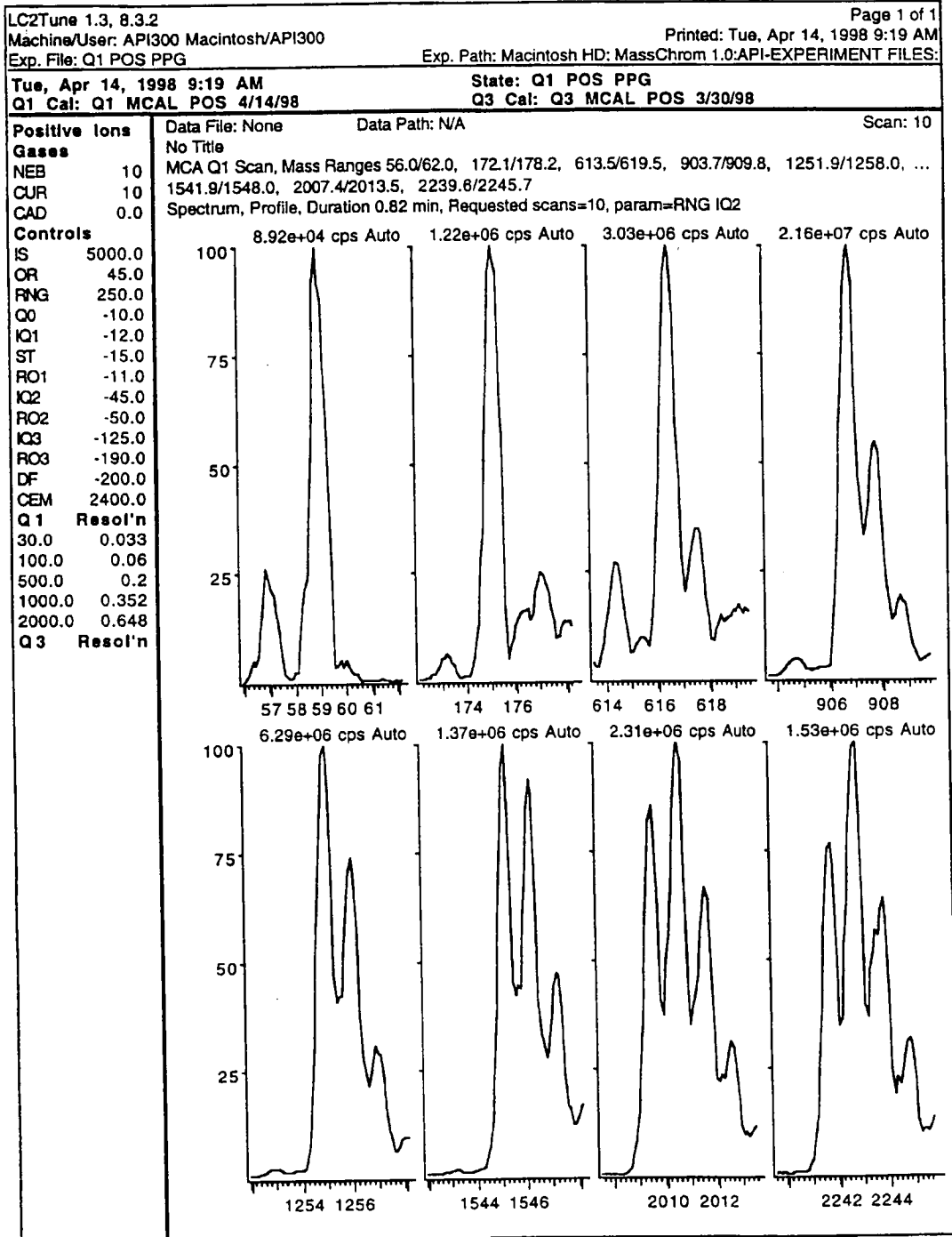


FIGURE 24 (continued)  
 Mass Calibration Printout (continued)

LC2Tune 1.3, 8.3.2			Page 1 of 1	
Machine/User: API300 Macintosh/API300			Printed: Tue, Apr 14, 1998 9:19 AM	
Exp. File: Q1 POS PPG			Exp. Path: Macintosh HD: MassChrom 1.0:API-EXPERIMENT FILES:	
Tue, Apr 14, 1998 9:19 AM			State: Q1 POS PPG	
Q1 Cal: Q1 MCAL POS 4/14/98			Q3 Cal: Q3 MCAL POS 3/30/98	
Reference Profile Data				
Search Range (amu)	2.0			
Threshold (icps)	10			
Width At (% max)	50			
Reference File: PPG Pos				
Scan 10			Q1 Scan	
Mass	Found At	$\Delta$ Mass	Inten	Width
59.0	58.992	-0.008	89200	0.71
175.133	175.121	-0.012	1223400	0.729
616.464	616.463	-0.001	3028500	0.743
906.673	906.699	0.026	21629300	0.72
1254.925	1254.914	-0.011	6289000	0.699
1545.134	1545.146	0.012	1373500	0.627
2010.469	2010.471	0.002	2305100	0.771
2242.637	2242.629	-0.008	1529000	0.776

FIGURE 25  
Typical LC/MS Chromatogram  
1.0 ng/mL Standard, 15 µL Injected, Set # 2-01-MV, Run # 68166

MacQuan, version 1.5

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Printed: Sun, Apr 19, 1998 22:00

Calibration File: CalibFile#3 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:

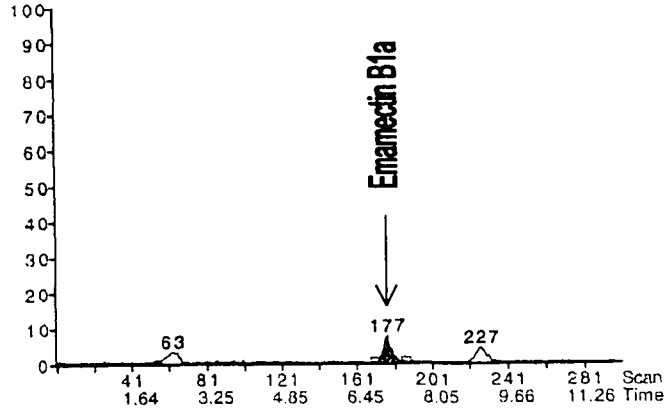
Comments: Run# 68166: 2-01-MV

d310 1.0 ng/ml Std. Fri, Apr 17, 1998 04:02  
No Comment

11.97 in 1 period  
Emamectin B1a  
No Internal Standard  
Use Area

intensity: 2500 cps

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 15.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 7.09  
Area 2084  
Height 186  
Start Time 6.85  
End Time 7.45  
Integration Width 0.60  
Retention Time 7.09  
Integration Type A - BB



d310 1.0 ng/ml Std. Fri, Apr 17, 1998 04:02  
No Comment

11.97 in 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area

Intensity: 2500 cps

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 10.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 9.10  
Area 1600  
Height 102  
Start Time 8.82  
End Time 9.50  
Integration Width 0.68  
Retention Time 9.10  
Integration Type A - BB

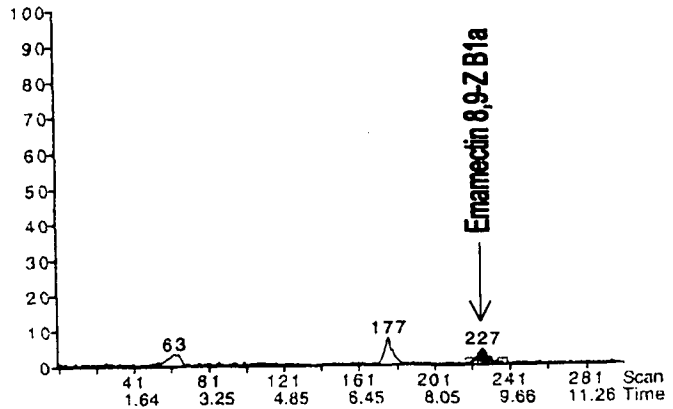


FIGURE 26  
Typical LC/MS Chromatogram  
2.0 ng/mL Standard, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68166

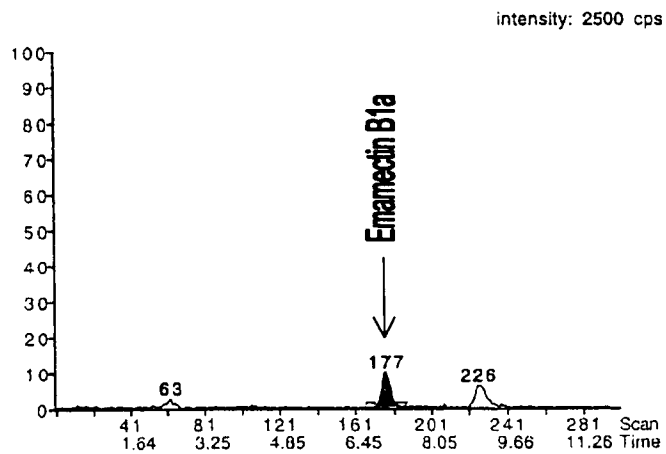
MacQuan, version 1.5  
Printed: Sun, Apr 19, 1998 22:00  
Calibration File: CalibFile#3 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:  
Comments: Run# 68166: 2-01-MV

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d314 2.0 ng/ml Std. Fri, Apr 17, 1998 04:57  
No Comment

11.97 in 1 period  
Emamectin B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 15.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 7.09  
Area 3108  
Height 249  
Start Time 6.77  
End Time 7.37  
Integration Width 0.60  
Retention Time 7.09  
Integration Type A - BV



d314 2.0 ng/ml Std. Fri, Apr 17, 1998 04:57  
No Comment

11.97 in 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 10.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 9.10  
Area 2660  
Height 152  
Start Time 8.85  
End Time 9.62  
Integration Width 0.76  
Retention Time 9.06  
Integration Type A - BB

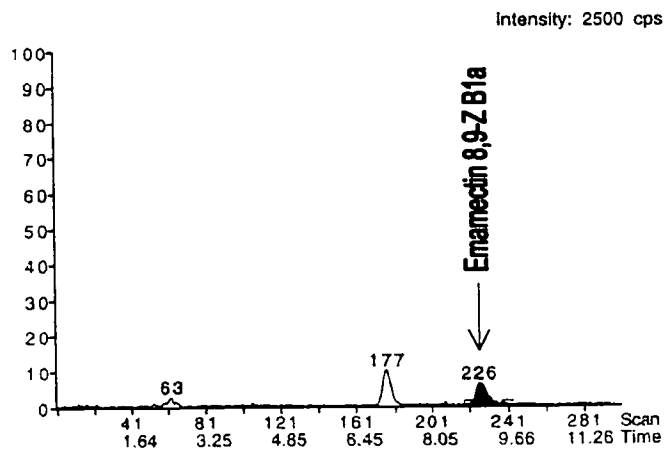


FIGURE 27  
Typical LC/MS Chromatogram  
5.0 ng/mL Standard, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68166

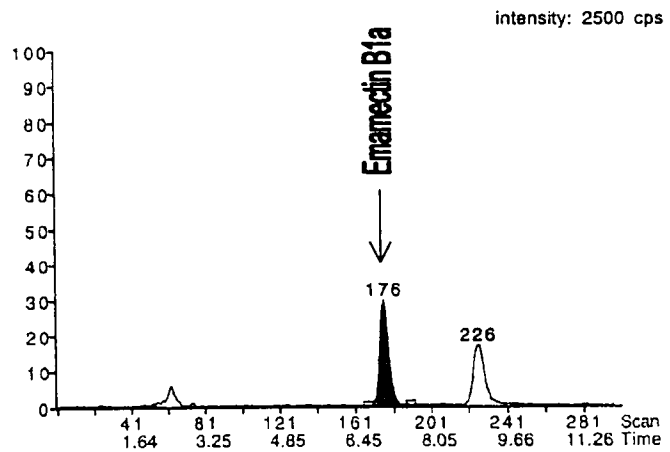
MacQuan, version 1.5  
Printed: Sun, Apr 19, 1998 22:00  
Calibration File: CalibFile#3 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:  
Comments: Run# 68166: 2-01-MV

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d318 5.0 ng/ml Std. Fri, Apr 17, 1998 05:51  
No Comment

11.97 in 1 period  
Emamectin B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 15.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 7.09  
Area 9509  
Height 746  
Start Time 6.73  
End Time 7.57  
Integration Width 0.84  
Retention Time 7.05  
Integration Type A - BB



d318 5.0 ng/ml Std. Fri, Apr 17, 1998 05:51  
No Comment

11.97 in 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 10.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 9.10  
Area 7730  
Height 425  
Start Time 8.73  
End Time 10.22  
Integration Width 1.48  
Retention Time 9.06  
Integration Type A - BB

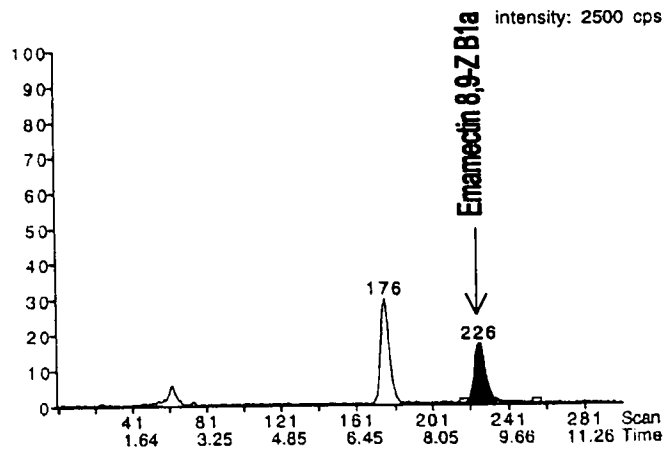


FIGURE 28  
Typical LC/MS Chromatogram  
8.0 ng/mL Standard, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68166

MacQuan, version 1.5

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Printed: Sun, Apr 19, 1998 22:00

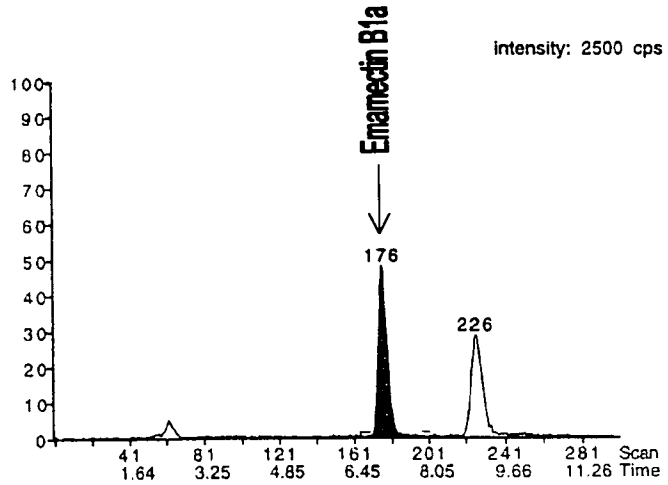
Calibration File: CalibFile#3 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:

Comments: Run# 68166: 2-01-MV

d321 8.0 ng/ml Std. Fri, Apr 17, 1998 06:32  
No Comment

11.97 in 1 period  
Enamectin B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 15.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 7.09  
Area 15666  
Height 1201  
Start Time 6.69  
End Time 7.93  
Integration Width 1.24  
Retention Time 7.05  
Integration Type A - BB



d321 8.0 ng/ml Std. Fri, Apr 17, 1998 06:32  
No Comment

11.97 in 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 10.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 9.10  
Area 11670  
Height 710  
Start Time 8.77  
End Time 9.70  
Integration Width 0.92  
Retention Time 9.06  
Integration Type A - BV

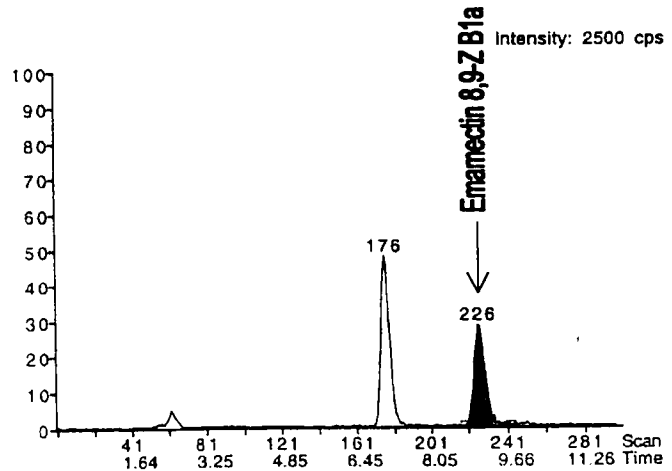


FIGURE 29  
Typical LC/MS Chromatogram  
10.0 ng/mL Standard, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68166

MacQuan, version 1.5

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Printed: Sun, Apr 19, 1998 22:00

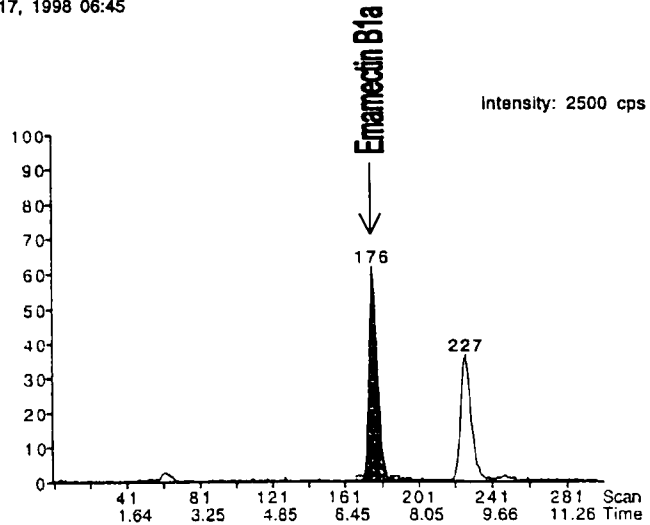
Calibration File: CalibFile#3 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:

Comments: Run# 68166: 2-01-MV

d322 10.0 ng/ml Std. Fri, Apr 17, 1998 06:45  
No Comment

11.97 in 1 period  
Emamectin B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 15.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 7.09  
Area 19074  
Height 1539  
Start Time 6.81  
End Time 7.49  
Integration Width 0.68  
Retention Time 7.05  
Integration Type A - BB



d322 10.0 ng/ml Std. Fri, Apr 17, 1998 06:45  
No Comment

11.97 in 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 10.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 9.10  
Area 14448  
Height 904  
Start Time 8.78  
End Time 9.70  
Integration Width 0.92  
Retention Time 9.10  
Integration Type A - BV

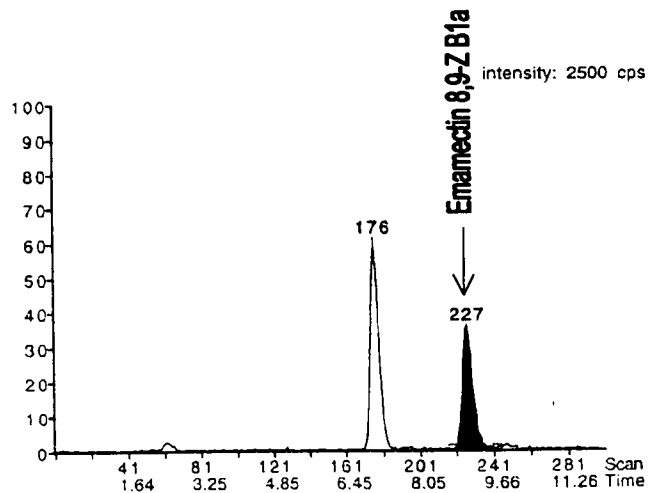


FIGURE 30  
Typical LC/MS Chromatogram  
0.523 ng/mL Standard, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68165

MacQuan, version 1.5  
Printed: Sun, Apr 19, 1998 22:06  
Calibration File: CalibFile#4 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:  
Comments: Run# 68165: 1-01-MV & 2-01-MV B1b

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d302 0.523 ng/ml Std. Fri, Apr 17, 1998 02:14  
No Comment

11.97 in 1 period  
Emamectin B1b  
No Internal Standard  
Use Area

intensity: 700 cps

1: 11.94 Q1 M1, 299 scans  
872.7  
Noise Thres. 4.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 20  
Smooth 0  
Expected RT 5.57  
Area 692  
Height 56  
Start Time 5.13  
End Time 5.73  
Integration Width 0.80  
Retention Time 5.57  
Integration Type A - BB

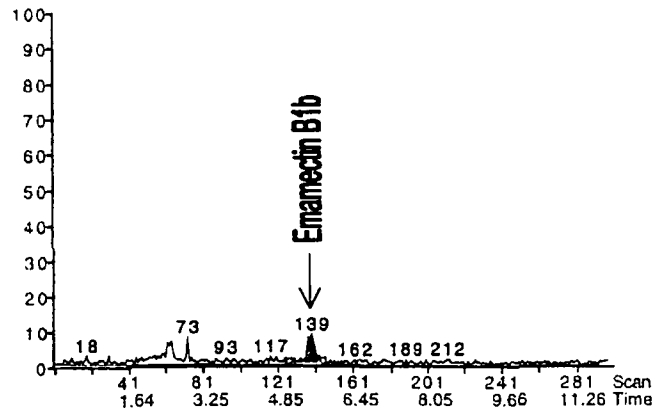


FIGURE 31  
Typical LC/MS Chromatogram  
1.046 ng/mL Standard, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68165

MacQuan, version 1.5

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Printed: Sun, Apr 19, 1998 22:06

Calibration File: CalibFile#4 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:

Comments: Run# 68165: 1-01-MV & 2-01-MV B1b

d305 1.046 ng/ml Std. Fri, Apr 17, 1998 02:54  
No Comment

11.97 in 1 period  
Emamectin B1b  
No Internal Standard  
Use Area

intensity: 700 cps

1: 11.94 Q1 M1, 299 scans  
872.7  
Noise Thres. 4.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 20  
Smooth 0  
Expected RT 5.57  
Area 1351  
Height 130  
Start Time 5.33  
End Time 5.85  
Integration Width 0.52  
Retention Time 5.57  
Integration Type A - BB

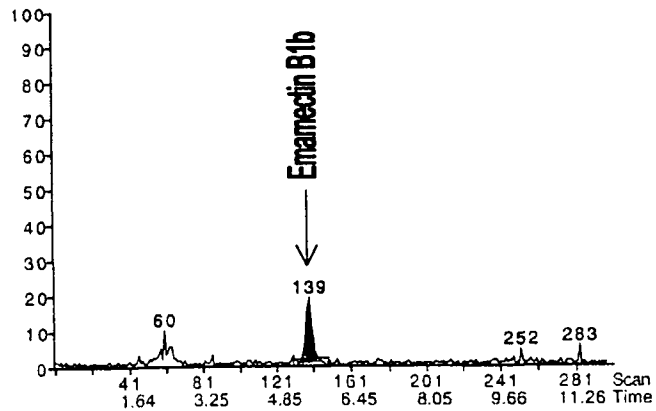


FIGURE 32  
Typical LC/MS Chromatogram  
2.61 ng/mL Standard, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68165

MacQuan, version 1.5

Page 1 of 9

Printed: Sun, Apr 19, 1998 22:06

Calibration File: CalibFile#4 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:

Comments: Run# 68165: 1-01-MV & 2-01-MV B1b

d301 2.61 ng/ml Std. Fri, Apr 17, 1998 02:00  
No Comment

11.97 In 1 period  
Emamectin B1b  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
872.7  
Noise Thres. 4.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 20  
Smooth 0  
Expected RT 5.57  
Area 2875  
Height 289  
Start Time 5.37  
End Time 5.81  
Integration Width 0.44  
Retention Time 5.57  
Integration Type A - VB

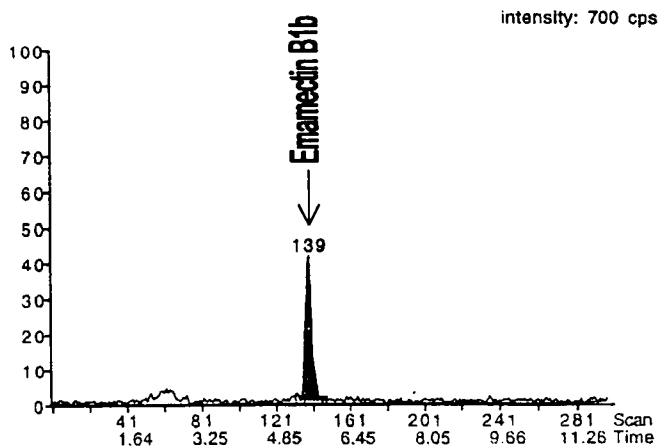


FIGURE 33  
Typical LC/MS Chromatogram  
4.184 ng/mL Standard, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68165

MacQuan, version 1.5

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Printed: Sun, Apr 19, 1998 22:06

Calibration File: CalibFile#4 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:

Comments: Run# 68165: 1-01-MV & 2-01-MV B1b

d308 4.184 ng/ml Std. No Comment

Fri, Apr 17, 1998 03:35

11.97 in 1 period  
Emamectin B1b  
No Internal Standard  
Use Area

Intensity: 700 cps

1: 11.94 Q1 M1, 299 scans  
872.7  
Noise Thres. 4.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 20  
Smooth 0  
Expected RT 5.57  
Area 4905  
Height 467  
Start Time 5.37  
End Time 6.05  
Integration Width 0.68  
Retention Time 5.53  
Integration Type A - VB

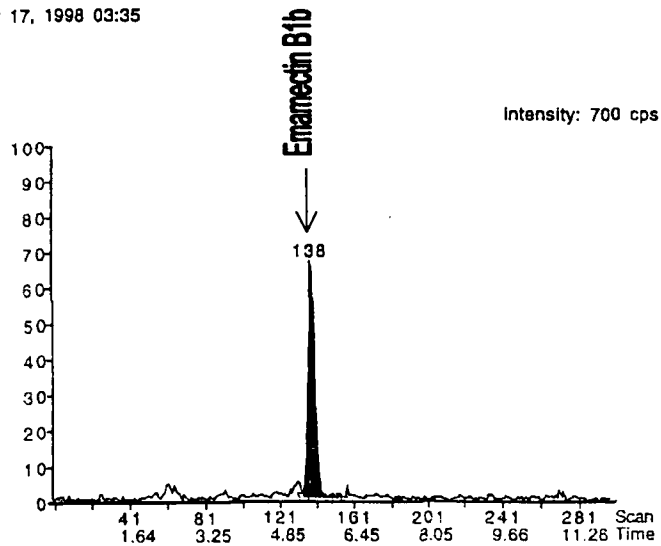


FIGURE 34  
Typical LC/MS Chromatogram  
5.23 ng/mL Standard, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68165

MacQuan, version 1.5

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Printed: Sun, Apr 19, 1998 22:06

Calibration File: CalibFile#4 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:

Comments: Run# 68165: 1-01-MV & 2-01-MV B1b

d309 5.23 ng/ml Std. Fri, Apr 17, 1998 03:49  
No Comment

11.97 in 1 period  
Emamectin B1b  
No Internal Standard  
Use Area

1: 11.94 Q1 M1, 299 scans  
872.7

Noise Thres. 4.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 20  
Smooth 0  
Expected RT 5.57

Area 6483  
Height 617  
Start Time 5.01  
End Time 5.77  
Integration Width 0.76  
Retention Time 5.49  
Integration Type A - BB

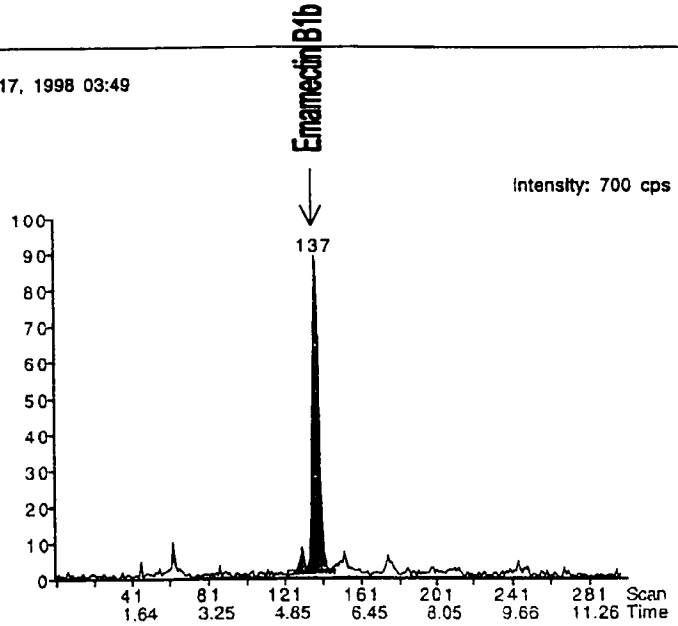


FIGURE 35  
 Typical LC/MS Calibration Curve (Emamectin B1a)  
 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68166

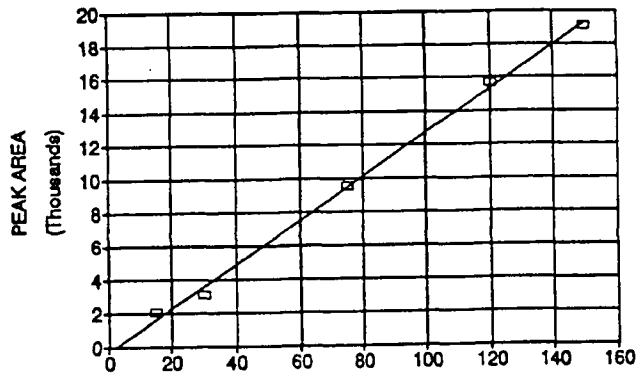
LLS rev.2.0  
 CALC/GRAPH  
 EN-CAS LABS  
 04/10/92 dis  
 ~~~~~

Proj.#: 98-0012      CalcDate: 05/13/98  
 Set: 2-01-MV      By(Init): ABR *BR 5-13-98*  
 Analyte: EMAMECTIN B1a      RunNumbr 68166

| X   | Y     |
|-----|-------|
| 15  | 2084  |
| 30  | 3108  |
| 75  | 9509  |
| 120 | 15666 |
| 150 | 19074 |

| STATISTICS          |          |            |
|---------------------|----------|------------|
| Regression Output:  |          |            |
| Constant            | -265.932 | (y-int)    |
| Std Err of Y Est    | 436.5327 |            |
| R Squared           | 0.997457 | (variance) |
| No. of Observations | 5        |            |
| Degress of Freedom  | 3        |            |
| X Coefficient(s)    | 130.1812 | (slope)    |
| Std Err of Coef.    | 3.79522  |            |
| Corr. Coeff.        | 0.998728 |            |

98-0012 EMAMECTIN B1a  
 2-01-MV BROCCOLI



WSP  
 5-20-98  
 Emu. G.

FIGURE 36  
 Typical LC/MS Calibration Curve (Emamectin B1b)  
 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68165

LLS rev.2.0  
 CALC/GRAPH  
 EN-CAS LABS  
 04/10/92 dis

Proj.#: 98-0012      CalcDate: 05/13/98  
 Set: 1-01-MV & 2-01-MV      By(Init): WEP  
 Analyte: EMAMECTIN B1b      RunNumbr 68165

*WEP*  
 5-13-98

| X     | Y    |
|-------|------|
| 39.15 | 2875 |
| 7.85  | 692  |
| 15.69 | 1351 |
| 62.76 | 4905 |
| 78.45 | 6483 |

| STATISTICS          |          |            |
|---------------------|----------|------------|
| Regression Output:  |          |            |
| Constant            | -14.2743 | (y-int)    |
| Std Err of Y Est    | 212.2908 |            |
| R Squared           | 0.994242 | (variance) |
| No. of Observations | 5        |            |
| Degrees of Freedom  | 3        |            |
| X Coefficient(s)    | 80.32061 | (slope)    |
| Std Err of Coef.    | 3.529026 |            |
| Corr. Coeff.        | 0.997117 |            |

98-0012 1-01-MV & 2-01-MV  
 EMAMECTIN LETTUCE & BROCCOLI

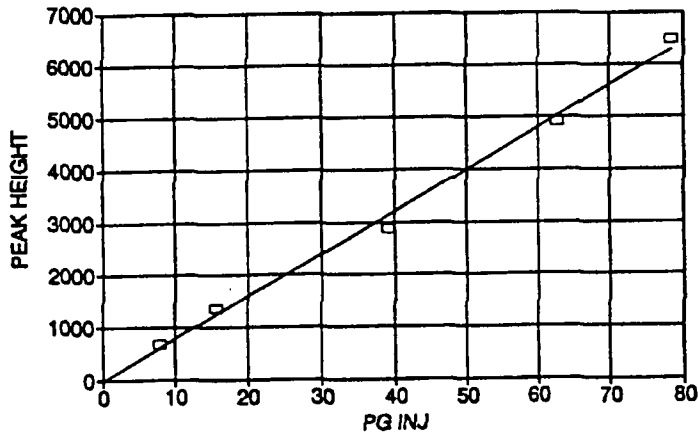


FIGURE 37  
 Typical LC/MS Calibration Curve (Emamectin 8,9-Z B1a)  
 15 µL Injected, Set # 2-01-MV, Run # 68166

LLS rev.2.0  
 CALC/GRAPH  
 EN-CAS LABS  
 04/10/92 dia  
 ~~~~~

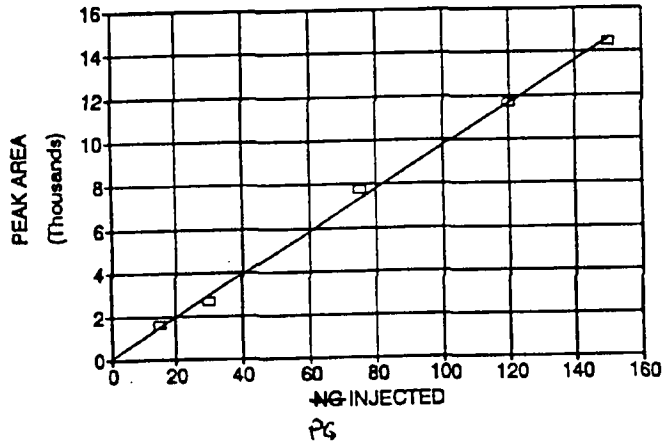
Proj.#: 98-0012  
 Set: 2-01-MV  
 Analyte: 8,9-Z B1a

CalcDate: 05/13/98  
 By(Init): ABR *ABR 5-13-98*  
 RunNumbr 68166

X	Y
15	1600
30	2660
75	7730
120	11670
150	14448

STATISTICS		
Regression Output:		
Constant	82.62585	(y-int)
Std Err of Y Est	308.1174	
R Squared	0.997701	(variance)
No. of Observations	5	
Degrees of Freedom	3	
X Coefficient(s)	96.65351	(slope)
Std Err of Coef.	2.678775	
Corr. Coeff.	0.99885	

98-0012 8,9-Z B1a  
 2-01-MV BROCCOLI



*WEP 5-20-98*  
*J. W. E.*

**FIGURE 38**  
 Typical LC/MS Chromatogram  
 Reagent Blank, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68166  
 EN-CAS Sample Code R. Blk

MacQuan, version 1.5  
 Printed: Sun, Apr 19, 1998 22:00  
 Calibration File: CallbFile#3 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:  
 Comments: Run# 68166: 2-01-MV

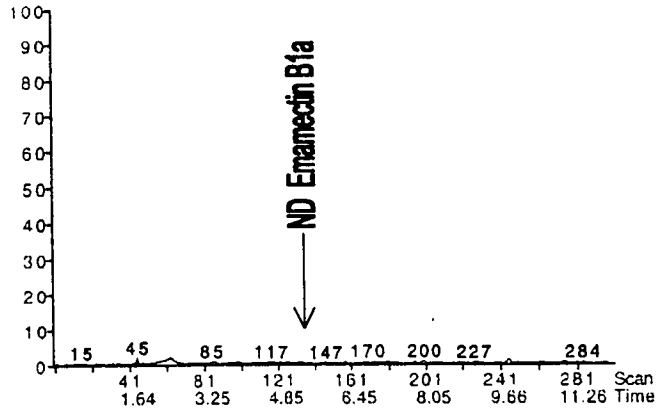
Page 2 of 13

d311 R. Blk. Fri, Apr 17, 1998 04:16  
 No Comment

11.97 in 1 period  
 Emamectin B1a  
 No Internal Standard  
 Use Area

Intensity: 2500 cps

1: 11.94 Q1 MI, 299 scans  
 886.7  
 Noise Thres. 15.0  
 Quant Thres. 1.5  
 Min. Width 6  
 Mult. Width 6  
 Base. Width 100  
 RT Win. (secs) 35  
 Smooth 0  
 Expected RT 7.09  
 Area 0  
 Height 0  
 Start Time -0.00  
 End Time -0.00  
 Integration Width -0.00  
 Retention Time -0.00  
 Integration Type



d311 R. Blk. Fri, Apr 17, 1998 04:16  
 No Comment

11.97 in 1 period  
 8,9-Z B1a  
 No Internal Standard  
 Use Area

Intensity: 2500 cps

1: 11.94 Q1 MI, 299 scans  
 886.7  
 Noise Thres. 10.0  
 Quant Thres. 1.5  
 Min. Width 6  
 Mult. Width 6  
 Base. Width 100  
 RT Win. (secs) 35  
 Smooth 0  
 Expected RT 9.10  
 Area 0  
 Height 0  
 Start Time -0.00  
 End Time -0.00  
 Integration Width -0.00  
 Retention Time -0.00  
 Integration Type

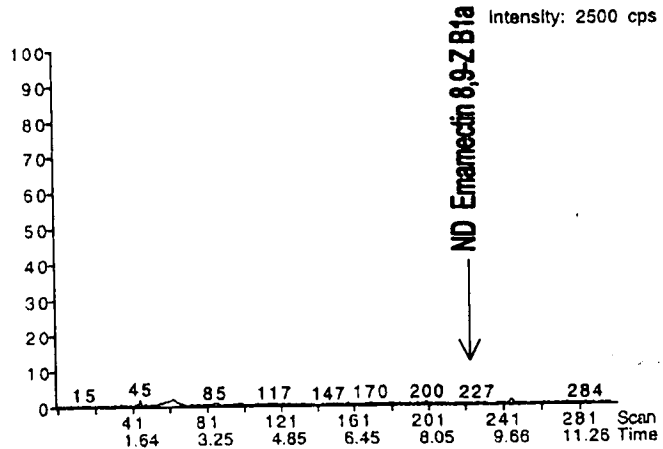


FIGURE 39  
Typical LC/MS Chromatogram  
Control Broccoli, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68166  
EN-CAS Sample Code EP4797-C2  
Quantitated for Emamectin B1a

MacQuan, version 1.5  
Printed: Sun, Apr 19, 1998 22:00  
Calibration File: CalibFile#3 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:  
Comments: Run# 68166: 2-01-MV

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d312 EP4797-C2 Fri, Apr 17, 1998 04:30  
No Comment

11.97 in 1 period  
Emamectin B1a  
No Internal Standard  
Use Area

Intensity: 2500 cps

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 15.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 8  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 7.09  
Area 0  
Height 0  
Start Time -0.00  
End Time -0.00  
Integration Width -0.00  
Retention Time -0.00  
Integration Type

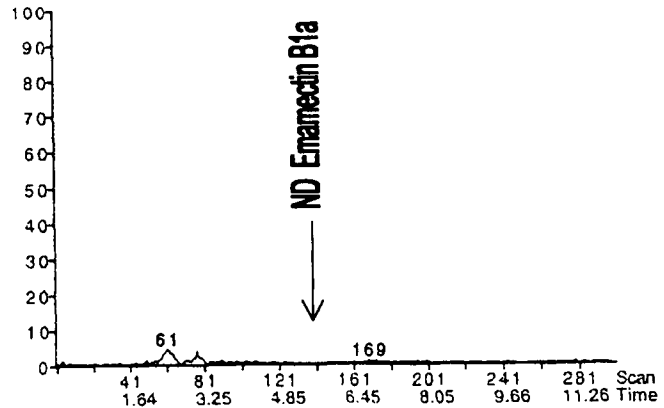


FIGURE 40  
Typical LC/MS Chromatogram  
Control Broccoli, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68165  
EN-CAS Sample Code EP4797-C2  
Quantitated for Emamectin B1b

MacQuan, version 1.5

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Printed: Sun, Apr 19, 1998 22:06

Calibration File: CallbFile#4 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:

Comments: Run# 68165: 1-01-MV & 2-01-MV B1b

d306 EP4797-C2 Fri, Apr 17, 1998 03:08  
No Comment

11.97 in 1 period  
Emamectin B1b  
No Internal Standard  
Use Area

intensity: 700 cps

1: 11.94 Q1 M1, 299 scans

872.7

Noise Thres.	4.0
Quant Thres.	1.5
Min. Width	6
Mult. Width	5
Base. Width	100
RT Win. (secs)	20
Smooth	0
Expected RT	5.57
Area	0
Height	0
Start Time	-0.00
End Time	-0.00
Integration Width	-0.00
Retention Time	-0.00
Integration Type	

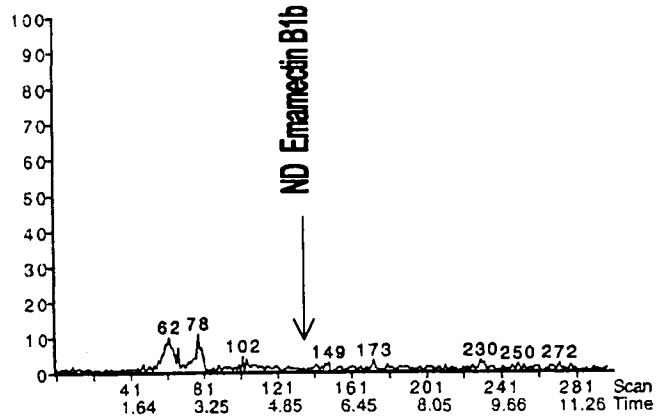


FIGURE 41  
Typical LC/MS Chromatogram  
Control Broccoli + 5 ppb MK-0244, 15  $\mu$ L Injected, Set # 2-03-MV,  
Run # 69504, EN-CAS Sample Code EP4797-S13

MacQuan, version 1.5  
Printed: Tue, May 12, 1998 09:35  
Calibration File: Untitled Path: N/A  
Comments: run #69504;2-03-mv

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d313 EP4797-S13 Mon, May 11, 1998 18:25  
No Comment

11.97 In 1 period  
Emamectin B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 2.4  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 5  
Bass. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 6.73  
Area 1730  
Height 146  
Start Time 6.29  
End Time 6.85  
Integration Width 0.56  
Retention Time 6.49  
Integration Type A - BB

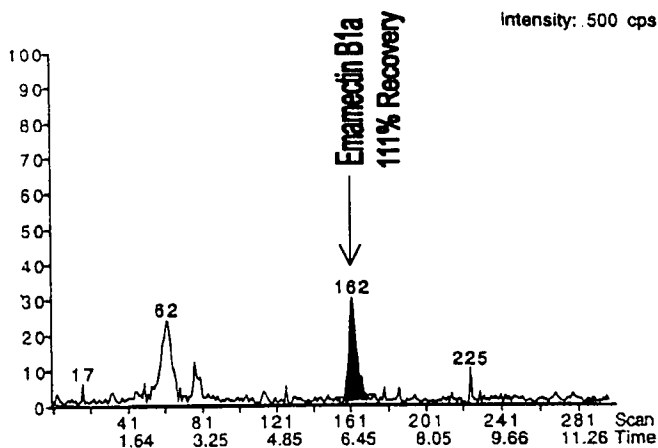


FIGURE 42  
Typical LC/MS Chromatogram  
Control Broccoli + 10 ppb MK-0244, 15  $\mu$ L Injected, Set # 2-02-MV,  
Run # 68191, EN-CAS Sample Code EP4797-S11

MacQuan, version 1.5  
Printed: Fri, May 1, 1998 09:45  
Calibration File: CalibFile 202md Path: Macintosh HD: MassChrom 1.0:98-0012:2-02-MV:  
Comments: run#68191; 2-02-mv

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d207 EP4797-S11 Thu, Apr 30, 1998 19:54  
No Comment

11.97 in 1 period  
Emamectin B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans

886.7

Noise Thres. 12.0

Quant Thres. 2.5

Min. Width 6

Mult. Width 5

Base. Width 100

RT Win. (secs) 35

Smooth 0

Expected RT 7.53

Area 24938

Height 2141

Start Time 7.01

End Time 7.69

Integration Width 0.68

Retention Time 7.29

Integration Type A - BV

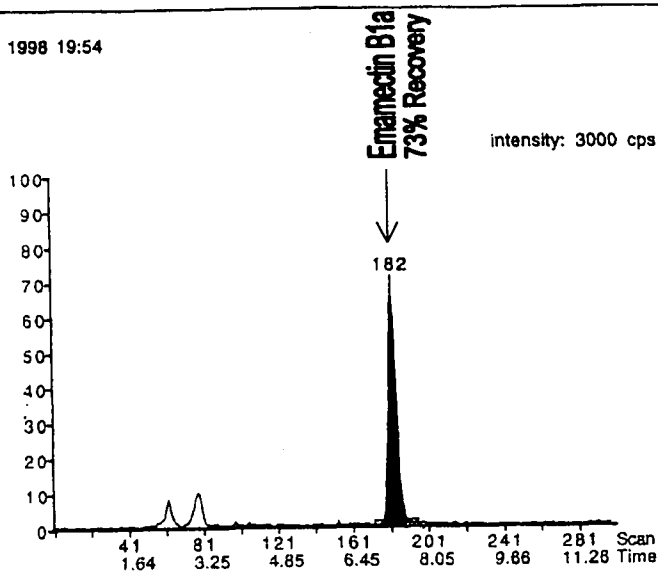


FIGURE 43  
Typical LC/MS Chromatogram  
Control Broccoli + 50 ppb MK-0244, 15  $\mu$ L Injected, Set # 2-01-MV, Run # 68166  
EN-CAS Sample Code EP4797-S7, 1 to 10 Dilution  
NOTE: See Figure 44 for undiluted injection for quantitation of Emamectin B1b.

MacQuan, version 1.5  
Printed: Sun, Apr 19, 1998 22:00  
Calibration File: CalibFile#3 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:  
Comments: Run# 68166: 2-01-MV

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d319 EP4797-S7 1:10 Fri, Apr 17, 1998 06:05  
No Comment

11.97 in 1 period  
Emamectin B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 M1. 299 scans  
886.7  
Noise Thres. 15.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 7.09  
Area 6825  
Height 561  
Start Time 6.49  
End Time 7.17  
Integration Width 0.68  
Retention Time 6.77  
Integration Type A - BB

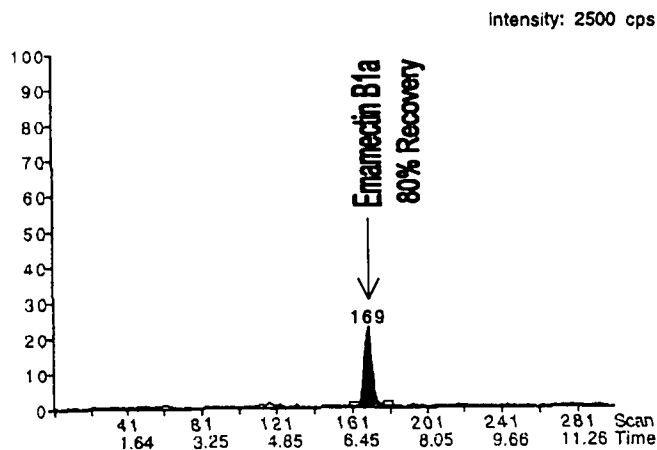


FIGURE 44  
Typical LC/MS Chromatogram  
Control Broccoli + 50 ppb MK-0244 (2.62 ppb B1b), 15  $\mu$ L Injected, Set # 2-01-MV,  
Run # 68165, EN-CAS Sample Code EP4797-S7

MacQuan, version 1.5  
Printed: Sun, Apr 19, 1998 22:06  
Calibration File: CalibFile#4 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:  
Comments: Run# 68165: 1-01-MV & 2-01-MV B1b

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d307 EP4797-S7 Fri, Apr 17, 1998 03:22  
No Comment

11.97 in 1 period  
Enamectin B1b  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
872.7  
Noise Thres. 4.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 20  
Smooth 0  
Expected RT 5.57  
Area 2410  
Height 228  
Start Time 5.33  
End Time 5.81  
Integration Width 0.48  
Retention Time 5.57  
Integration Type A - VB

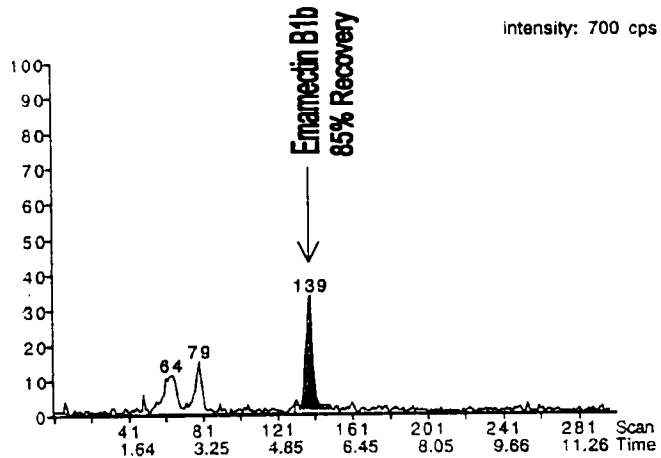


FIGURE 45  
Typical LC/MS Chromatogram  
Control Lettuce, 15  $\mu$ L Injected, Set # 1-01-MV, Run # 68154  
EN-CAS Sample Code EP4796-C2  
Quantitated for B1a

MacQuan. version 1.5

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Printed: Mon, Apr 13, 1998 14:36

Calibration File: CallbFile#1 Path: Macintosh HD: MassChrom 1.0:98-0025:1-01-MV:

Comments: Run#68154: 1-01-MV

d102 EP4796-C2 Fri, Apr 10, 1998 17:23  
No Comment

11.97 in 1 period  
Emamectin B1a  
No Internal Standard  
Use Area

intensity: 2000 cps

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 12.0  
Quant Thres. 2.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 6.97  
Area 0  
Height 0  
Start Time -0.00  
End Time -0.00  
Integration Width -0.00  
Retention Time -0.00  
Integration Type

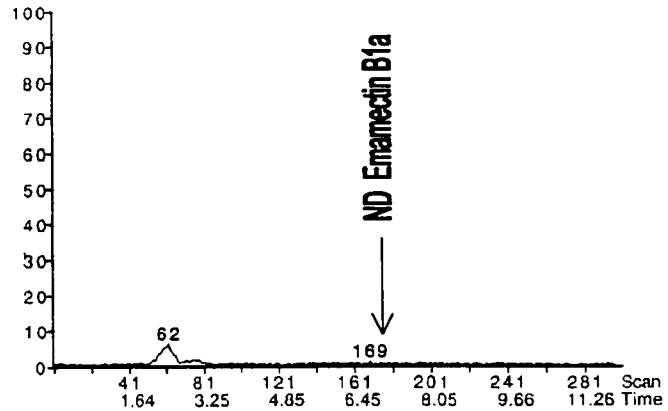


FIGURE 46  
Typical LC/MS Chromatogram  
Control Lettuce, 15  $\mu$ L Injected, Set # 1-01-MV, Run # 68165  
EN-CAS Sample Code EP4796-C2  
Quantitated for Emamectin B1b

MacQuan, version 1.5

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Printed: Sun, Apr 19, 1998 22:06

Calibration File: CallbFile#4 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:

Comments: Run# 68165: 1-01-MV & 2-01-MV B1b

d303 EP4796-C2 Fri, Apr 17, 1998 02:27  
No Comment

11.97 in 1 period  
Emamectin B1b  
No Internal Standard  
Use Area

intensity: 700 cps

1: 11.94 Q1 M1, 299 scans  
872.7  
Noise Thres. 4.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 20  
Smooth 0  
Expected RT 5.57  
Area 0  
Height 0  
Start Time -0.00  
End Time -0.00  
Integration Width -0.00  
Retention Time -0.00  
Integration Type

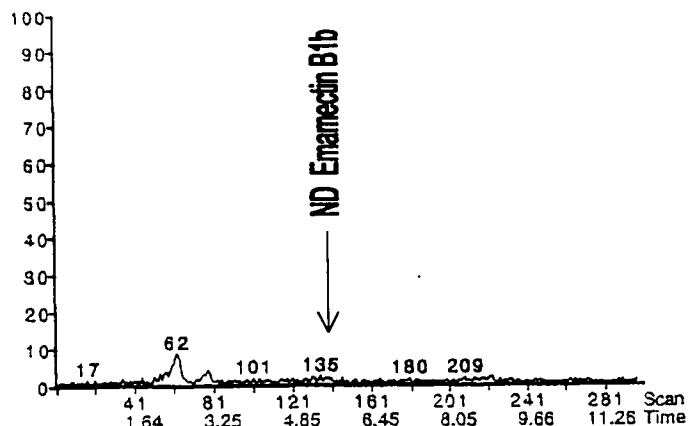


FIGURE 47  
Typical LC/MS Chromatogram  
Control Lettuce, 15  $\mu$ L Injected, Set # 1-01-MV, Run # 68154  
EN-CAS Sample Code EP4796-C2  
Quantitated for Emamectin 8,9-Z B1a

d102 EP4796-C2 Fri, Apr 10, 1998 17:23  
No Comment

11.97 in 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 M1, 299 scans  
886.7  
Noise Thres. 10.0  
Quant Thres. 2.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 8.97  
Area 0  
Height 0  
Start Time -0.00  
End Time -0.00  
Integration Width -0.00  
Retention Time -0.00  
Integration Type

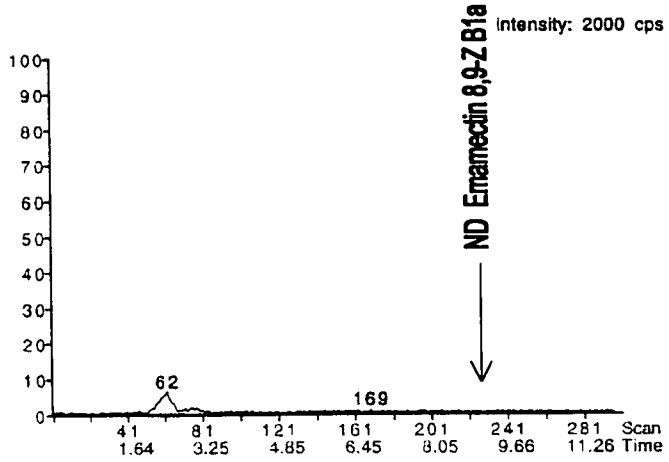


FIGURE 48  
Typical LC/MS Chromatogram  
Control Lettuce + 5 ppb Emamectin 8,9-Z B1a, 15  $\mu$ L Injected, Set # 1-01-MV  
Run # 68154, EN-CAS Sample Code EP4796-S4

d104 EP4796-S4 Fri, Apr 10, 1998 17:50  
No Comment

11.97 In 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 10.0  
Quant Thres. 2.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 8.97  
Area 6372  
Height 385  
Start Time 8.69  
End Time 9.58  
Integration Width 0.88  
Retention Time 8.93  
Integration Type A - BB

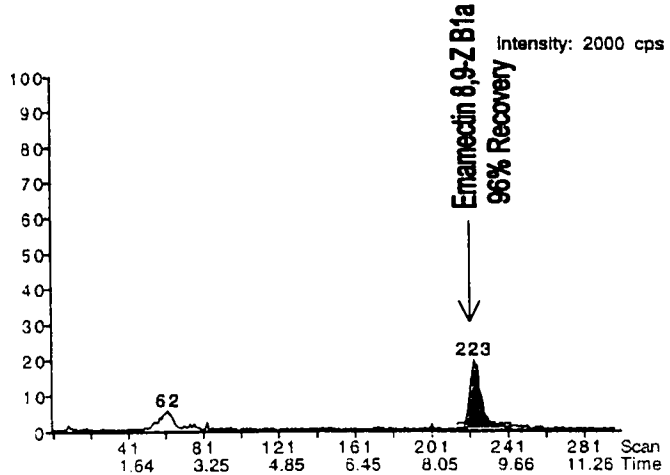


FIGURE 49  
Typical LC/MS Chromatogram  
Control Lettuce + 10 ppb Emamectin 8,9-Z B1a, 15  $\mu$ L Injected, Set # 1-01-MV  
Run # 68154, EN-CAS Sample Code EP4796-S6

d107 EP4796-S6 Fri, Apr 10, 1998 18:31  
No Comment

11.97 in 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area  
1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 10.0  
Quant Thres. 2.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 8.97  
Area 12548  
Height 780  
Start Time 8.69  
End Time 9.58  
Integration Width 0.88  
Retention Time 9.02  
Integration Type A - BV

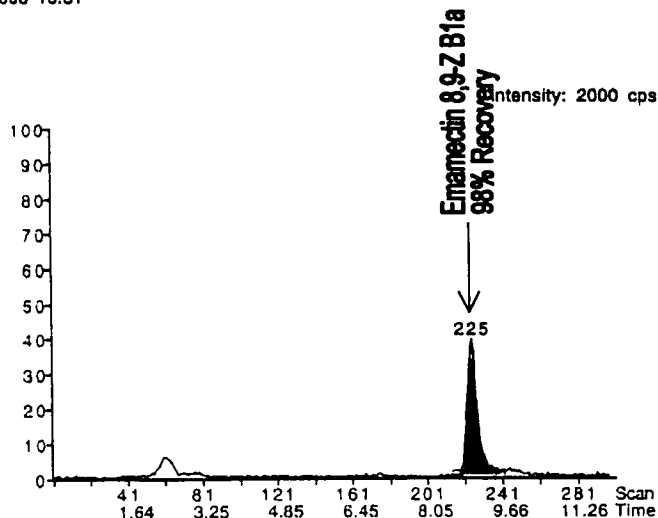


FIGURE 50  
Typical LC/MS Chromatogram  
Control Lettuce + 50 ppb Emamectin B1a, 15  $\mu$ L Injected, Set # 1-01-MV  
Run # 68154, EN-CAS Sample Code EP4796-S7, 1 to 10 Dilution

MacQuan, version 1.5  
Printed: Mon, Apr 13, 1998 14:37  
Calibration File: CalibFile#1 Path: Macintosh HD: MassChrom 1.0:98-0025:1-01-MV:  
Comments: Run#68154: 1-01-MV

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d110 EP4796-S7 1:10 Fri, Apr 10, 1998 19:12  
No Comment

11.97 in 1 period  
Emamectin B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 M1. 299 scans  
886.7  
Noise Thres. 12.0  
Quant Thres. 2.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 6.97  
Area 7496  
Height 592  
Start Time 6.81  
End Time 7.45  
Integration Width 0.64  
Retention Time 7.01  
Integration Type A - BV

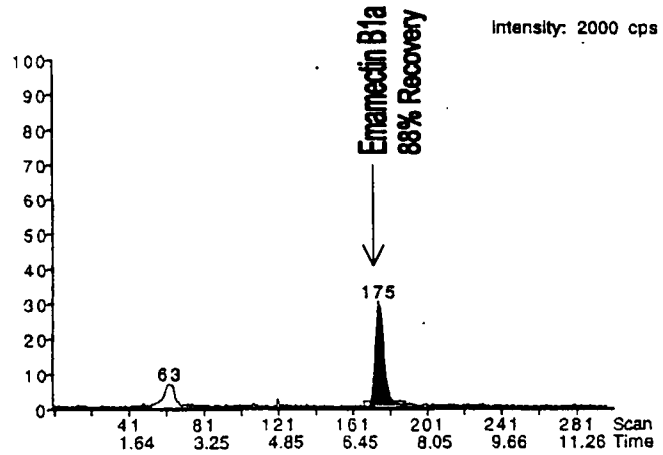


FIGURE 51  
Typical LC/MS Chromatogram  
Control Lettuce + 50 ppb MK-0244 (2.62 ppb B1b), 15  $\mu$ L Injected, Set # 1-01-MV  
Run # 68165, EN-CAS Sample Code EP4796-S7

MacQuan, version 1.5  
Printed: Sun, Apr 19, 1998 22:08  
Calibration File: CalibFile#4 Path: Macintosh HD: MassChrom 1.0:98-0012:2-01-MV:  
Comments: Run# 68165: 1-01-MV & 2-01-MV B1b

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d304 EP4796-S7 Fri, Apr 17, 1998 02:41  
No Comment

11.97 In 1 period  
Emamectin B1b  
No Internal Standard  
Use Area

1: 11.94 Q1 M1, 299 scans  
872.7  
Noise Thres. 4.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 5  
Base. Width 100  
RT Win. (secs) 20  
Smooth 0  
Expected RT 5.57  
Area 2773  
Height 271  
Start Time 5.33  
End Time 5.81  
Integration Width 0.48  
Retention Time 5.48  
Integration Type A - VV

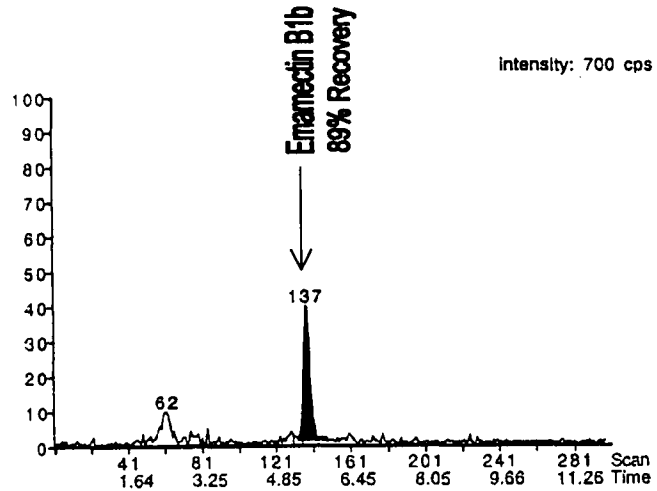


FIGURE 52  
Typical LC/MS Chromatogram  
Control Celery, 15  $\mu$ L Injected, Set # 3-01-MV, Run # 68178  
EN-CAS Sample Code EP4798-C2

d416 EP4798-C2 Wed, Apr 22, 1998 22:07  
No Comment

11.97 In 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area  
1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 15.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 9.14  
Area 0  
Height 0  
Start Time -0.00  
End Time -0.00  
Integration Width -0.00  
Retention Time -0.00  
Integration Type

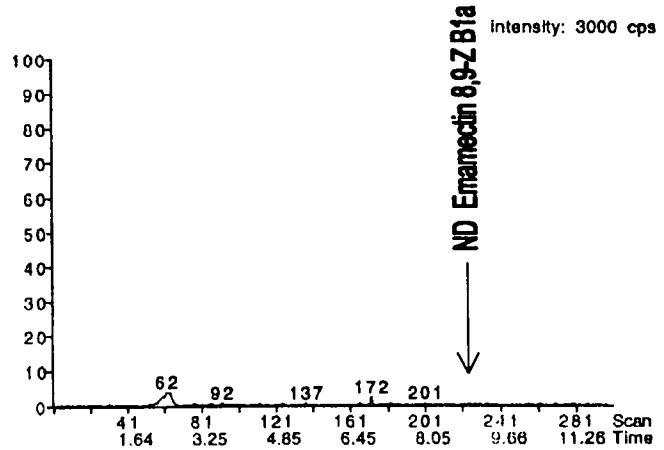


FIGURE 53  
Typical LC/MS Chromatogram  
Control Celery + 5 ppb Emamectin 8,9-Z B1a, 15  $\mu$ L Injected, Set # 3-01-MV  
Run # 68178, EN-CAS Sample Code EP4798-S4

d419 EP4798-S4 Wed, Apr 22, 1998 22:47  
No Comment

11.97 in 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 15.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 9.14  
Area 9300  
Height 667  
Start Time 8.69  
End Time 9.58  
Integration Width 0.88  
Retention Time 9.02  
Integration Type A - BB

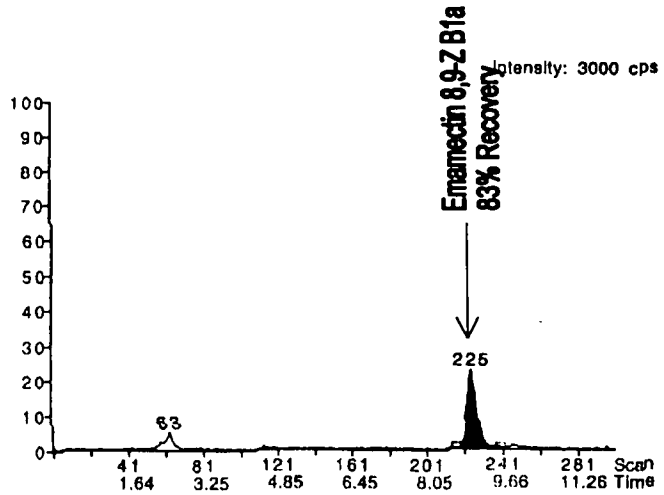


FIGURE 54  
Typical LC/MS Chromatogram  
Control Celery + 15 ppb Emamectin 8,9-Z B1a, 15  $\mu$ L Injected, Set # 3-01-MV  
Run # 68178, EN-CAS Sample Code EP4798-S6, 1 to 2 Dilution

d421 EP4798-S6 1:2  
No Comment

Wed, Apr 22, 1998 23:15

11.97 in 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans

886.7

Noise Thres. 15.0

Quant Thres. 1.5

Mln. Width 6

Mult. Width 6

Base. Width 100

RT Wln. (secs) 35

Smooth 0

Expected RT 9.14

Area 14561

Height 974

Start Time 8.73

End Time 10.02

Integration Width 1.28

Retention Time 9.06

Integration Type A - BB

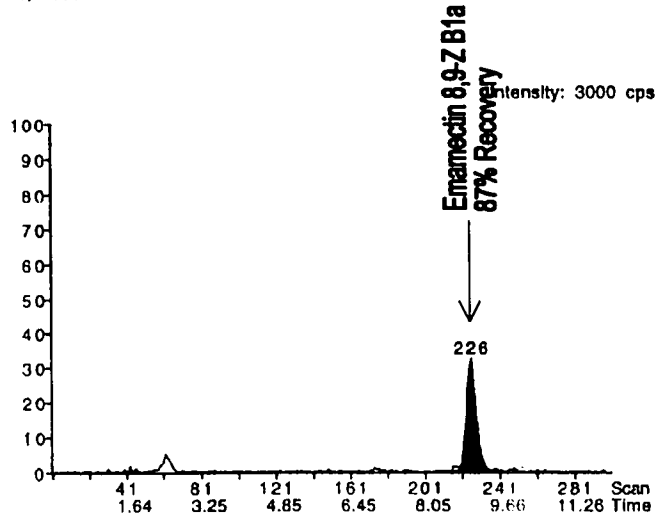


FIGURE 55  
Typical LC/MS Chromatogram  
Control Celery + 50 ppb Emamectin 8,9-Z B1a, 15  $\mu$ L Injected, Set # 3-01-MV  
Run # 68178, EN-CAS Sample Code EP4798-S6, 1 to 10 Dilution

d424 EP4798-S6 1:10 Wed, Apr 22, 1998 23:55  
No Comment

11.97 in 1 period  
8,9-Z B1a  
No Internal Standard  
Use Area

1: 11.94 Q1 MI, 299 scans  
886.7  
Noise Thres. 15.0  
Quant Thres. 1.5  
Min. Width 6  
Mult. Width 6  
Base. Width 100  
RT Win. (secs) 35  
Smooth 0  
Expected RT 9.14  
Area 9235  
Height 628  
Start Time 8.69  
End Time 9.62  
Integration Width 0.92  
Retention Time 8.06  
Integration Type A - BV

