

**AMERICAN CYANAMID COMPANY
AGRICULTURAL PRODUCTS RESEARCH DIVISION
ENVIRONMENTAL SCIENCES
P.O. BOX 400
PRINCETON, NEW JERSEY 08543-0400**

Recommended Method of Analysis - M 2686

CL 303,630 (chlorphenapyr): GC Determinative and GC/MS Confirmatory Method for the Determination of CL 303,630 Residues in Various Fruits (such as Stone Fruits, Pome Fruits, Strawberries and Grapes)

A. Principle

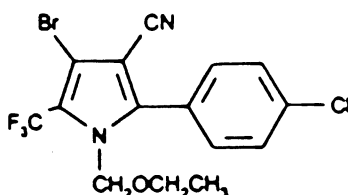
Residues of CL 303,630 are extracted from samples with methanol/Milli-Q water and purified using solid phase extraction techniques. Quantitation of CL 303,630 residues is accomplished by fused silica capillary gas chromatography equipped with an electron capture detector. Results are calculated as CL 303,630 by the direct comparison of peak heights to those of external standards. The validated sensitivity (LOQ, limit of quantitation) of this method is 0.05 ppm for each commodity.

CL 303,630 residues can be confirmed by using GC/mass spectrometry (chemical ionization mode) and selected ion monitoring at m/z 347⁻ and 349⁻.

B. Reagents (Items from other manufacturers may be used, if they are **proven** to be functionally equivalent.)

1. Analytical Standard: CL 303,630, analytical grade of known purity. Obtained from American Cyanamid Company, Agricultural Products Research Division, P.O. Box 400, Princeton, New Jersey, 08543-0400.

CL 303,630: 4-bromo-2-(4-chlorophenyl)-1-(ethoxy-methyl)-5-(trifluoromethyl)-1H-pyrrole-3-carbonitrile.



M. W. = 407.6

2. **Solvents:** B & J Brand High Purity Solvents, Baxter Healthcare Corporation, Muskegon, MI.

- | | |
|-----------------------|------------|
| a. Methanol | c. Hexane |
| b. Methylene Chloride | d. Acetone |

3. **Water, Deionized:** Water passed through Millipore's Milli-Q Plus Ultra Pure Water System. Use this water for all steps.

4. **Solution:** Extraction Solvent (15% Water in Methanol): Dilute 150 mL of Milli-Q water (B.3) to 1 liter with methanol in a 1 liter volumetric flask and mix well.

C. **Apparatus** (Items from other manufacturers may be used, provided they are **proven** to be functionally equivalent.)

1. **Balance:** Analytical, Sartorius, precision ± 0.05 mg.
2. **Laboratory Glassware:** General laboratory.
3. **Flash Evaporator:** Buchler Instruments, Model RE-0121C equipped with a heated water bath maintained at approximately 30°C, and a dry ice solvent trap.
4. **Gas Chromatograph:** A Hewlett Packard Model 5890 Series II instrument equipped with an inlet system for a capillary column and a Nickel-63 High Temperature Electron Capture detector.
5. **Fused Silica Capillary Column:** 15 m x 0.53 mm nominal I.D.. SPB-20 bonded phase with film thickness of 1.0 micron, Supelco, Inc., Bellefonte, PA, Catalog Number 2-5332.

6. Recorder: Hewlett Packard, HP 3396A Integrator. (Must use real ink type recorders for GLP chromatogram long term storage.)
7. Plastic Syringe, Disposable: Luer-Lok, 30 mL capacity, Catalog Number 309662, Becton Dickinson & Co., Franklin Lakes, NJ.
8. Adaptors: IST (International Sorbent Technology) Isolute PTFE Column Adaptors, Distributed by Jones Chromatography, Lakewood, CO, Catalog Number 120-1100. (NOTE: Some adaptors from other suppliers may contain interfering contaminants which may give an EC response.)
9. Visiprep Solid Phase Extraction Manifold: Visiprep 24, Catalog Number 5-7250, distributed by Supelco, Inc., Bellefonte, PA.
10. Solid Phase Extraction Cartridges:
 - a. International Sorbent Technology (IST) Isolute C-18 Cartridges (1g/6 mL), Catalog Number 220-0100-C, distributed by Jones Chromatography, Lakewood, CO.
 - b. International Sorbent Technology (IST) Isolute Silica Cartridge (1g/6 mL), Catalog Number 460-0100-C, distributed by Jones Chromatography, Lakewood, CO.
11. Reservoir, Empty: 70-mL capacity, Catalog Number 120-1008 distributed by Jones Chromatography, Lakewood, CO.
12. Waring Blender: Model 31BL46 with 1-quart capacity glass blender jar. Waring Products Division, Dynamic Corp. of America, New Hartford, CT.
13. PowerStat Variable Auto Transformer: Input 120V, Output 0-140V, Catalog Number 62546-455, VWR Scientific Products Corporation, McGaw Park, IL .
14. Filter Paper: 9-cm diameter, glass fiber, 934-AH, Whatman Laboratory Division, Springfield Mill, Maidstone, Kent, England.
15. Filtering Flasks: 500-mL capacity, Corning Glass Works.
16. Filtering Funnel: Buchner, porcelain, 9-cm diameter.
17. Microliter Syringe: Hamilton, Model 701, 10- μ L capacity.
18. Food Chopper: Hobart, Model 84185-D.

19. Mass Spectrometer: Finnigan Mat SSQ710.
20. Gas Chromatograph: Varian Model 3400.
21. GC Column: 15m x 0.25 mm, 0.25 micron DB-5MS film (J&W Scientific, Cat. No 122-5512).

D. Preparation of Standard Solutions (Prepare monthly, store in amber bottles in refrigerator.)

1. Stock Solution

Weigh accurately a known amount (approximately 10 mg) of CL 303,630 into a 100-mL volumetric flask. Dilute to the mark with acetone and mix well. Calculate and record the exact concentration of CL 303,630.

NOTE: Resulting concentration of the standard stock solution must be corrected for standard purity.

2. Fortification and Gas Chromatographic Solutions

- a. Pipet into a 100-mL volumetric flask an appropriate amount of the Standard Stock Solution to deliver 1000 μg of CL 303,630. Dilute to the mark with acetone and mix well. This solution contains 10 $\mu\text{g/mL}$ of CL 303,630.
- b. Pipet into a 100-mL volumetric flask a 10-mL aliquot of the 10 $\mu\text{g/mL}$ standard solution. Dilute to the mark with acetone and mix well. This solution contains 1 $\mu\text{g/mL}$ of CL 303,630.
- c. Pipet into a 100-mL volumetric flask a 10-mL aliquot of the 1 $\mu\text{g/mL}$ standard solution. Dilute to the mark with hexane and mix well. This solution contains 0.1 $\mu\text{g/mL}$ of CL 303,630.
- d. Pipet into separate 100-mL volumetric flasks 2-, 1-, 0.5- and 0.25-mL aliquots of the 1 $\mu\text{g/mL}$ standard solution. Dilute to the mark with hexane and mix well. These solutions contain 0.02, 0.01, 0.005 and 0.0025 $\mu\text{g/mL}$ respectively, of CL 303,630 and are used for the linearity check. Use the 0.01 $\mu\text{g/mL}$ solution as the working standard for both GC determinative and GC/MS Confirmatory analyses.

E. Gas Chromatographic Conditions

Operating conditions described below are provided as a guide to establish actual operating conditions and should be adjusted as necessary to obtain CL 303,630 peak shape and resolution from background peaks equivalent to or better than those shown in Figures 1 to 6.

1. Instrument: Hewlett Packard Model 5890 Series II gas chromatograph.
2. Detector: Nickel-63 High Temperature Electron Capture Detector.
3. Column: Fused silica capillary, 15 m x 0.53 mm I.D., SPB-20 with film thickness 1.0 micron.
4. Integrator: HP 3396A.
5. Instrument Conditions:
 - a. Carrier Gas (Helium) 9.3 mL/min
 - b. Make-up Gas for Capillary Column (95% Argon/5% Methane) 67 mL/min
 - c. Chart Speed 0.5 cm/min
 - d. Retention Time approx. 6.1 minutes
 - e. Run Time approx. 10 minutes
 - f. Injection Volume 1 μ L
 - g. Injector Temperature 260°C
 - h. Detector Temperature 350°C
 - i. Initial Column Temperature 195°C
 - j. Initial Time (Hold) 0 minutes
 - k. Temperature Rate 7°C per minute
 - l. Final Temperature 240°C (Hold for 5 min)
 - m. Purge Time 0.50 min
 - n. Purge Flow 15 mL/min

If late-eluting peaks which might interfere with subsequent injections are a problem, then the following gradient programming is recommended.

- a. Initial Temperature 195°C
- b. Initial Time (Hold) 0 minutes
- c. Temperature Rate 7°C per minute
- d. Final Temperature 290°C
- e. Purge Time 0.50 min
- f. Hold 290°C for 5 minutes
- g. Injector Temperature 290°C

F. Linearity Check

1. Adjust the GC conditions to obtain a peak response of approximately 30-40% FSD (full-scale deflection) for a 10-pg injection of working standard (0.01 µg/mL).
2. Inject 1 µL of the standard solutions containing 0.0025, 0.005, 0.01 and 0.02 µg/mL.
3. Determine the response factor (ratio) for all injections by dividing the peak response by the amount (pg) injected. Calculate the average response ratio. A deviation of any standard response ratio by more than 15% from the average response ratio indicates instrumental or standard difficulties which must be corrected before proceeding with the analyses.
4. Linearity checks should be performed at least weekly during the analysis of samples from every field residue study and when the chromatographic system has been adjusted or serviced.

G. Sample Preparation

1. Pulverize sufficient dry ice in a food chopper (Hobart, Model 84185-D) to chill the bowl and blade thoroughly.
2. Add the prefrozen samples in small portions to enable reduction to fine particle size. It may be necessary to add small portions of dry ice during the chopping procedure to ensure that the samples remain in a frozen state.
3. Mix the sample well to obtain a good homogeneous mixture and pack an aliquot in several suitable containers.
4. Allow the frozen samples to stand in a freezer overnight for the dry ice to dissipate completely.
5. Keep all samples frozen until ready for analysis.

H. Recovery Test

The validity of the procedure should always be demonstrated by recovery tests before the analysis of unknown samples is attempted. As a quality control measure, at least one concurrent recovery should be run with each set of samples.

1. Weigh a 10- or 20-g sample of finely chopped commodities (see extraction steps for exact procedure) into an appropriate container and transfer to a 1-quart blender jar.
2. Add by pipet a volume of standard fortification solution appropriate to the fortification level to be tested.
3. Continue with the extraction and cleanup steps.

I. Extraction (all commodities)

1. Weigh 10 or 20 grams of the desired sample commodity according to Table I. Weigh into an appropriate container and transfer to a 1-quart blender jar.

Table I

<u>Commodity</u>	<u>Weight Taken, g</u>	<u>Aliquot for Analysis, mL</u>	<u>Final Volume, mL</u>
Stone Fruits: (Plums, Prunes and Cherries)	20	15	10
Peaches:	10	75	25
Pome Fruits: (Pears and Apples)	20	75	25
Strawberries:	20	15	10
Grapes:	20	15	10

2. Add 300 mL of extraction solvent to the blender jar and blend for approximately five minutes at moderate-high speed without splashing solvent out of the blender jar. Control blender speed with a Powerstat Variable Auto Transformer, if necessary.
3. Pass 5-10 mL of extraction solvent (Reagent B.4.) through a 9-cm glass-fiber filter paper held on a Buchner funnel using vacuum and a 500-mL filter flask. Discard the solution. Filter the extract from step I.2. using mild vacuum, collecting the filtrate in the 500-mL filtration flask.
4. Pipet a 15- or 75-mL sample aliquot (see Table I) into a 100- or 200-mL beaker. Add 20 mL of Milli-Q water to the 15-mL aliquot and 75 mL of Milli-Q water to the 75 mL aliquot. Mix thoroughly by gentle swirling. Proceed with step J.1 for all commodities except peaches.

5. For peaches pipet a 75-mL sample aliquot into a 250-mL separatory funnel. Add 30 mL of Milli-Q water to the separatory funnel. Partition the sample by vigorously shaking for 30 seconds with 100 mL of hexane. Repeat this step two times, collect and combine the upper clear hexane layers in a 500-mL evaporation flask. (The lower aqueous layer as well as the boundary layer should be collected in a beaker and then transferred quantitatively to the separatory funnel. Only the upper clean hexane layer is transferred to the 500-mL evaporation flask.) Evaporate the hexane layers to dryness using a flash evaporator. Dissolve the residue in 75 mL of 15% Milli-Q water/methanol followed by 75 mL of Milli-Q water. Proceed to step J.1.

J. Solid Phase Extraction Cleanup

1. Prepare an IST C-18 cartridge using a Visiprep Solid Phase Extraction Manifold. Wash the cartridge with 5 mL of methanol followed by 10 mL of Milli-Q water.
2. Connect a 70-mL non-fritted reservoir onto the top of the C-18 cartridge using an adaptor. Using vacuum, pull the sample from step I.4 or I.5 through the C-18 cartridge at an approximate rate of 1-2 drops/sec. Using vacuum, air dry the C-18 cartridge for 30 seconds (do not exceed).
3. Remove the 70-mL reservoir and C-18 cartridge from the Visiprep Solid Phase Extraction Manifold. Connect a 30-mL disposable syringe onto the top of the C-18 cartridge. Add 10 mL of hexane to the syringe and push the solution dropwise into a 100-mL evaporation flask using vacuum (1 drop/sec). Evaporate the hexane to dryness using a flash evaporator. (Use 15-30 mL of methanol to aid in water removal, if needed.)
4. Dissolve the residue in hexane according to Table I in Section I. For peaches proceed to step J. 5.
5. For peaches, dissolve the residue in 10 mL of methylene chloride. Prepare an IST silica gel cartridge using a Visiprep Solid Phase Extraction Manifold. Wash the cartridge with 5 mL of methylene chloride. Remove the cartridge from the Visiprep Solid Phase Extraction Manifold. Connect a 30-mL disposable syringe onto the top of the silica cartridge using an adaptor. Using vacuum, pass the 10-mL extract through the silica cartridge at the rate of 1-2 drops/second and collect in a 100-mL evaporation flask. Rinse the flask with 5 mL of methylene chloride and pass through the silica cartridge and collect in the same 100-mL evaporation flask. Evaporate the methylene chloride to dryness using a flash evaporator. Add 15 mL of methanol to the flask and evaporate to dryness using a flash evaporator.
6. Dissolve the residue in 25 mL of hexane for GC analysis.

K. Gas Chromatographic Analysis

1. After obtaining a stable GC response as described in Section F, inject a 1- μ L aliquot of the working standard (0.01 μ g/mL), followed by a maximum of two sample injections and another 1- μ L standard injection.
2. Compare the sample peak height with that obtained from a 10-pg injection of the GC working standard (1 μ L of the 0.01 μ g/mL working standard).
3. If the sample peak is higher than the highest linearity standard injected, dilute to an appropriate volume with hexane and reinject.
4. Make a standard injection after each sample or after every second sample and use the average peak height of the standard injection before and after the sample injection for the calculation.

L. Calculations

For each sample calculation, use the sample peak height and the average peak height measurement of the external standard obtained before and after the sample injection as follows:

$$\text{PPM} = \frac{R(\text{SAMP}) \times V1 \times V3 \times C(\text{STD}) \times V5 \times D.E.}{R(\text{STD}) \times W \times V2 \times V4}$$

$$\% \text{ RECOVERY} = \frac{\text{PPM FOUND} \times 100}{\text{FV} \times \text{FC}/W} = \frac{\text{PPM FOUND}}{\text{PPM ADDED}} \times 100$$

Where:

R(SAMP) = Peak height of the sample in millimeters

R(STD) = Average peak height of the working standards in millimeters

W = Weight of sample taken for analysis in grams (10 or 20 g)

V1 = Volume of extraction solvent used in milliliters (300 mL)

V2 = Aliquot of the extract taken for analysis in milliliters (15 or 75 mL)

- V3 = Volume of hexane added to dissolve final residues for chromatographic analysis in milliliters (10 mL or 25 mL)
- V4 = Volume of sample solution injected in microliters (1 μ L)
- V5 = Volume of working standard solution injected in microliters (1 μ L)
- C(STD) = Concentration of working standard solution injected in micrograms per milliliter (0.01 μ g/mL)
- D.F. = Dilution factor
- FV = Fortification volume in milliliters
- FC = Fortification concentration (of standard solution added) in micrograms per milliliter

Typical chromatograms for determining CL 303,630 residues in various fruit commodities are shown in Figures 1 through 6.

M. GC/MS Confirmatory Analysis

1. Sample Preparation for GC/MS Confirmation: Any commodities requiring mass spectrometric confirmation are directly amenable to mass spectrometric analysis.
2. GC/MS Standard Solution: Use the 0.01 μ g/mL (10 ng/mL) standard solution.
3. GC/MS Instrumentation: (Items from other manufacturers may be used provided they have been proven to be functionally equivalent.)
 - a. Mass Spectrometer: Finnigan MAT SSQ710
 - b. Gas Chromatograph: Varian Model 3400
 - c. GC Column: 5 m x 0.25 mm I.D., 0.25 micron DB-5MS film (J & W Scientific, Custom Number 115786)
4. GC/MS Conditions:
 - a. GC Column Oven Temperature 60°C for 0.5 min;
20°C/min to 280°C, hold at 280°C
for 5 min.
 - b. Injector Temperature 280°C (split valve open at 0.5 min.)
 - c. Transfer Line Temperature 250°C

d.	Column Head Pressure	5 psi H ₂
e.	Source Temperature	150°C
f.	CI Reagent Gas	Methane at 8200 mT (indicated)
g.	Conversion Dynode	+15 kV
h.	Electron Multiplier	-1200 Volts
i.	Preamplifier Range (Full Scan)	1 E-07 amps/volt
j.	Preamplifier Range (SIM)	1 E-08 amps/volt
k.	Ions Monitored	m/z 347 ⁻ , 349 ⁻
l.	Retention Time of Analyte	approximately 6.2 min.
m.	Mode Used	Negative Ion, Chemical Ionization
n.	Full Scan Range	300 to 400 m/z
o.	Scan Time	0.5 sec
p.	Injection Volume	1 µL

The conditions above are specific for the instruments on which they were determined. Conditions will vary from instrument to instrument and should be adjusted to give sensitivity and adequate resolution of well defined peaks at approximately the retention time listed in M.4.1. Prior to analysis, the mass spectrometer should be tuned to give proper resolution and peak shape on an appropriate reference material and the data system should be calibrated.

5. GC/MS Confirmatory Analysis:

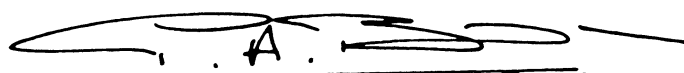
- a. Using parameters detailed in M.4, a 100-pg on-column injection of the analyte (1 µL of 0.1 µg/mL) should give a background-subtracted mass spectrum similar to that shown in Figure 7. Determine the mass centroids of the ions at m/z 347⁻ and 349⁻ and set the mass spectrometer for selected ion monitoring of these ions with a +/- 0.2 dalton scan window and a dwell time of 250 msec/ion (0.5 sec/scan).
- b. Inject 1-µL aliquots of the working standard (M.2) until a reasonably constant response is obtained (Figure 8).
- c. Follow the injection sequence working standard, Sample Number 1, Sample Number 2, working standard, Sample Number 3, Sample Number 4, working standard.
- d. If the response of the working standard decreases to an unacceptable level during the analysis, instrumental parameters should be adjusted to restore adequate sensitivity. If such adjustments are made, inject duplicate aliquots of the working standard to determine the new response values of the standard.

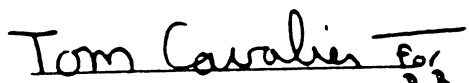
6. Data Treatment: The sample is confirmed as containing residues of CL 303,630 when:

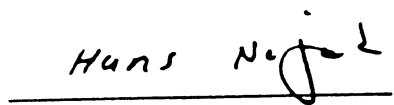
- a. The retention time of the presumed analyte in the sample is within 15 seconds of the averaged retention times of the analyte peaks in the bracketing standards.
- b. The ions indicative of the analyte in the sample maximize at retention times which are within 5 seconds of each other.
- c. The ratio of the m/z 347⁻ ion to the m/z 349⁻ ion in the sample agrees with that observed for the standard.
- d. The quantitation by GC/MS is comparable to the residue found by the determinative method.

Typical mass chromatograms for mass spectrum, standard, control and fortified samples are shown in Figures 9 through 18.

APPROVALS:


 Phillip Miller *for PM.* 06/11/97
 Sr. Group Leader Date
 Residue Chemistry


 Tom Cavalieri *for B.B.* 6-11-97
 Brion W. Babbitt Date
 Author


 Huns Nejad 6/11/97
 Author Date

/ct

Figure 1: Typical Chromatograms for the Determination of CL 303,630 Residues in Peaches

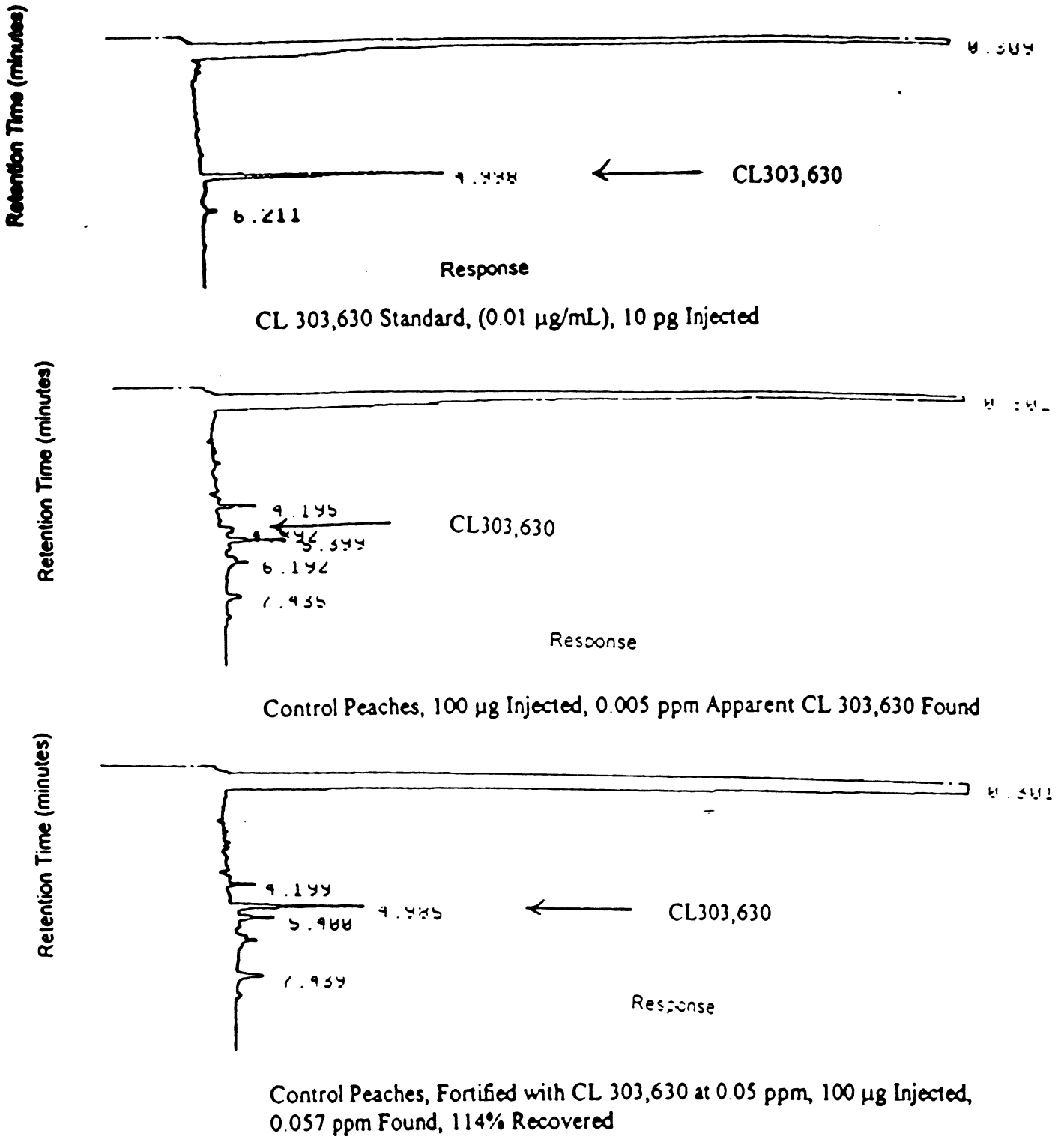


Figure 2: Typical Chromatograms for the Determination of CL 303,630 Residues in Plums

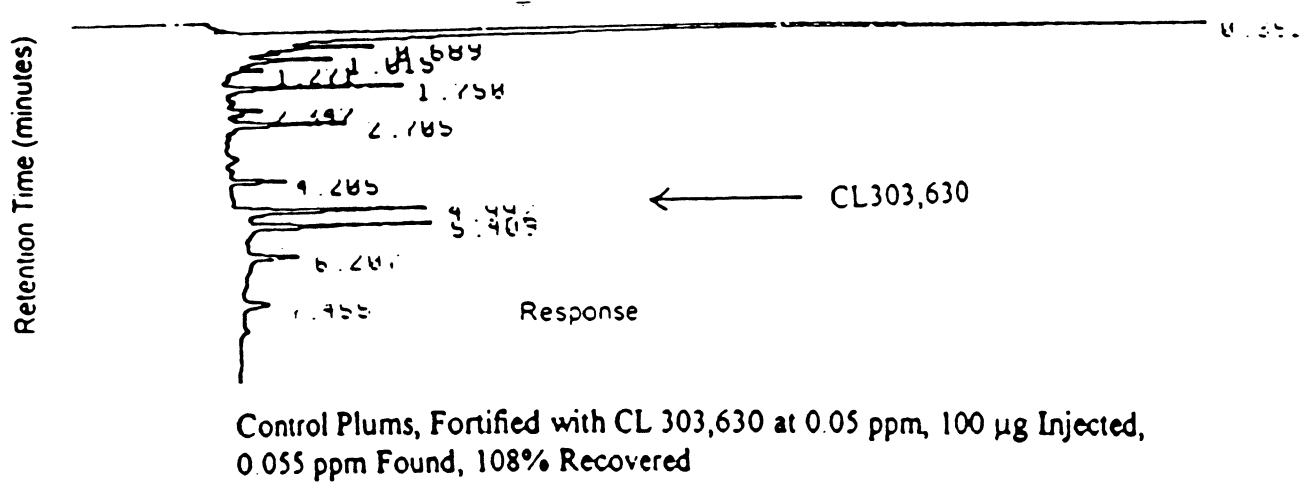
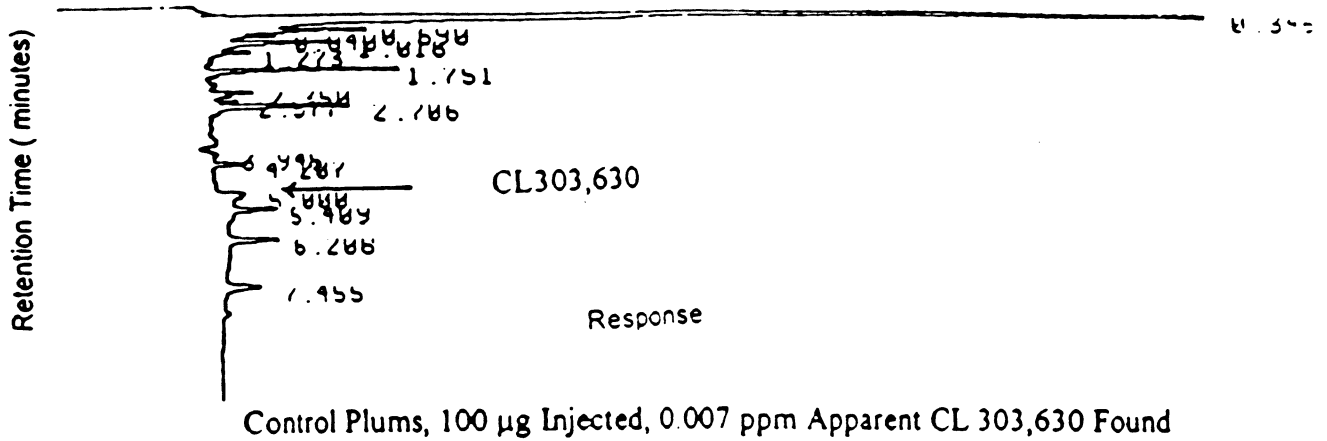
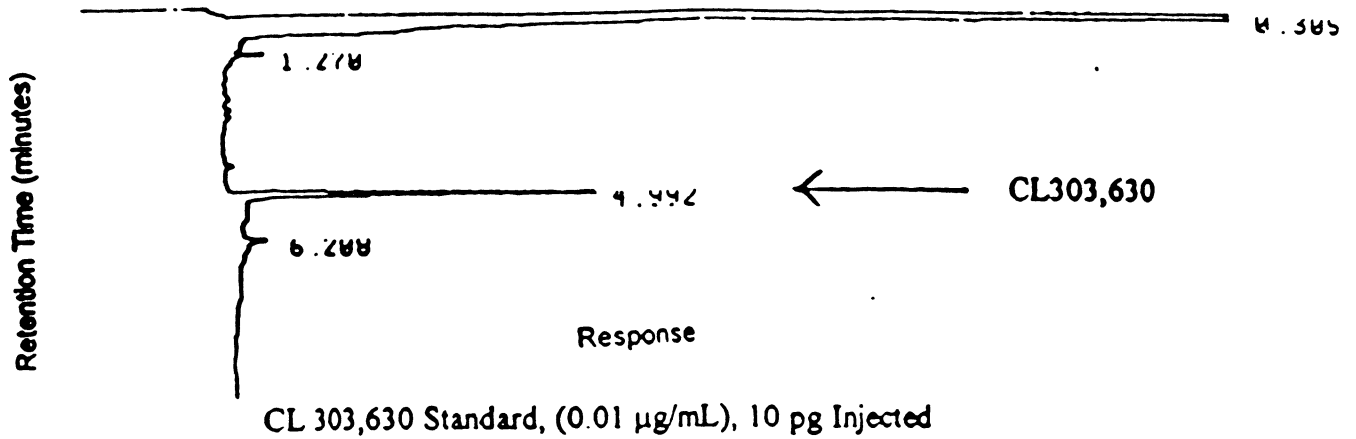


Figure 4: Typical Chromatograms for the Determination of CL 303,630 Residues in Red Apples

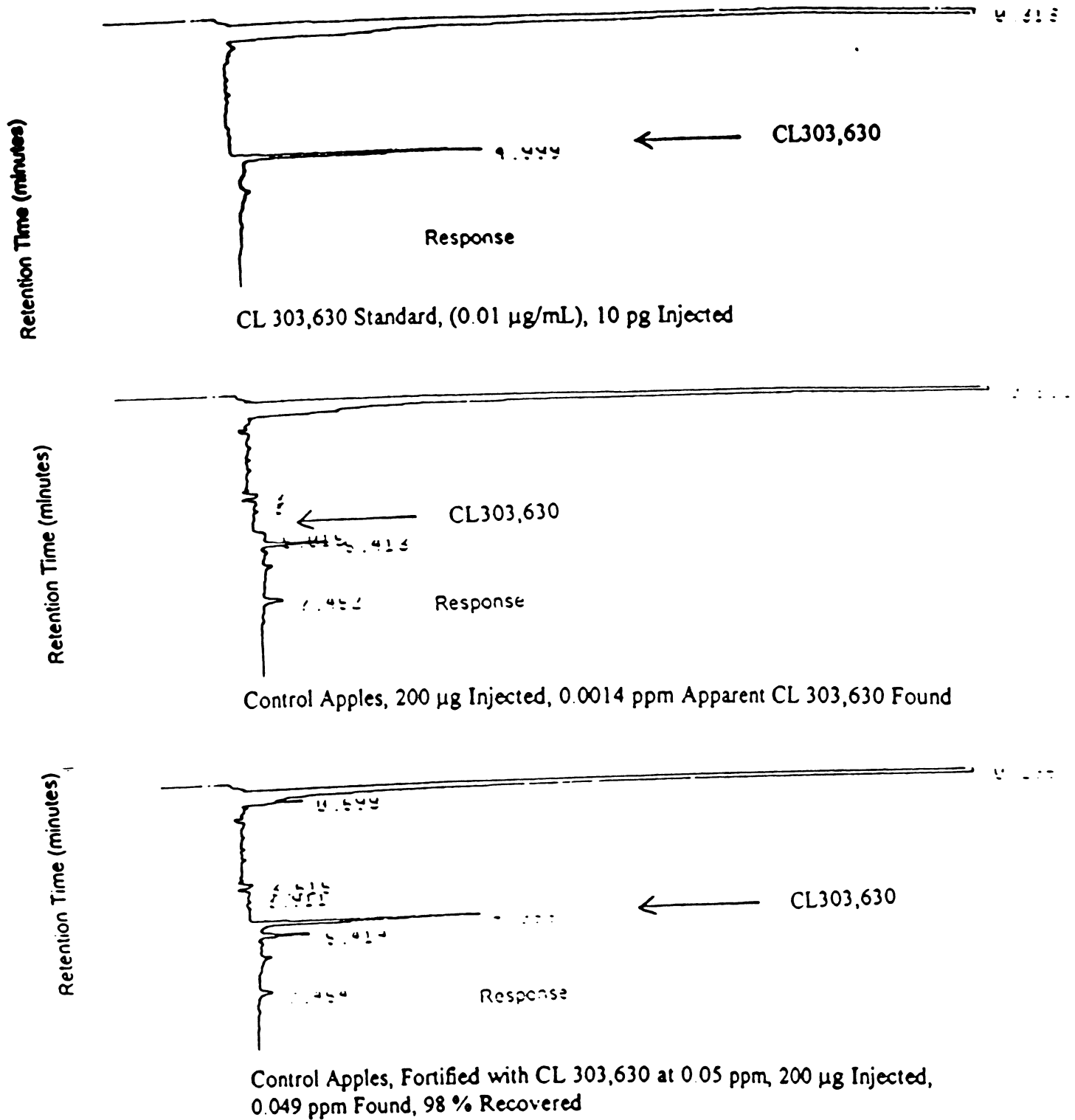


Figure 5: Typical Chromatograms for the Determination of CL 303,630 Residues in Strawberries

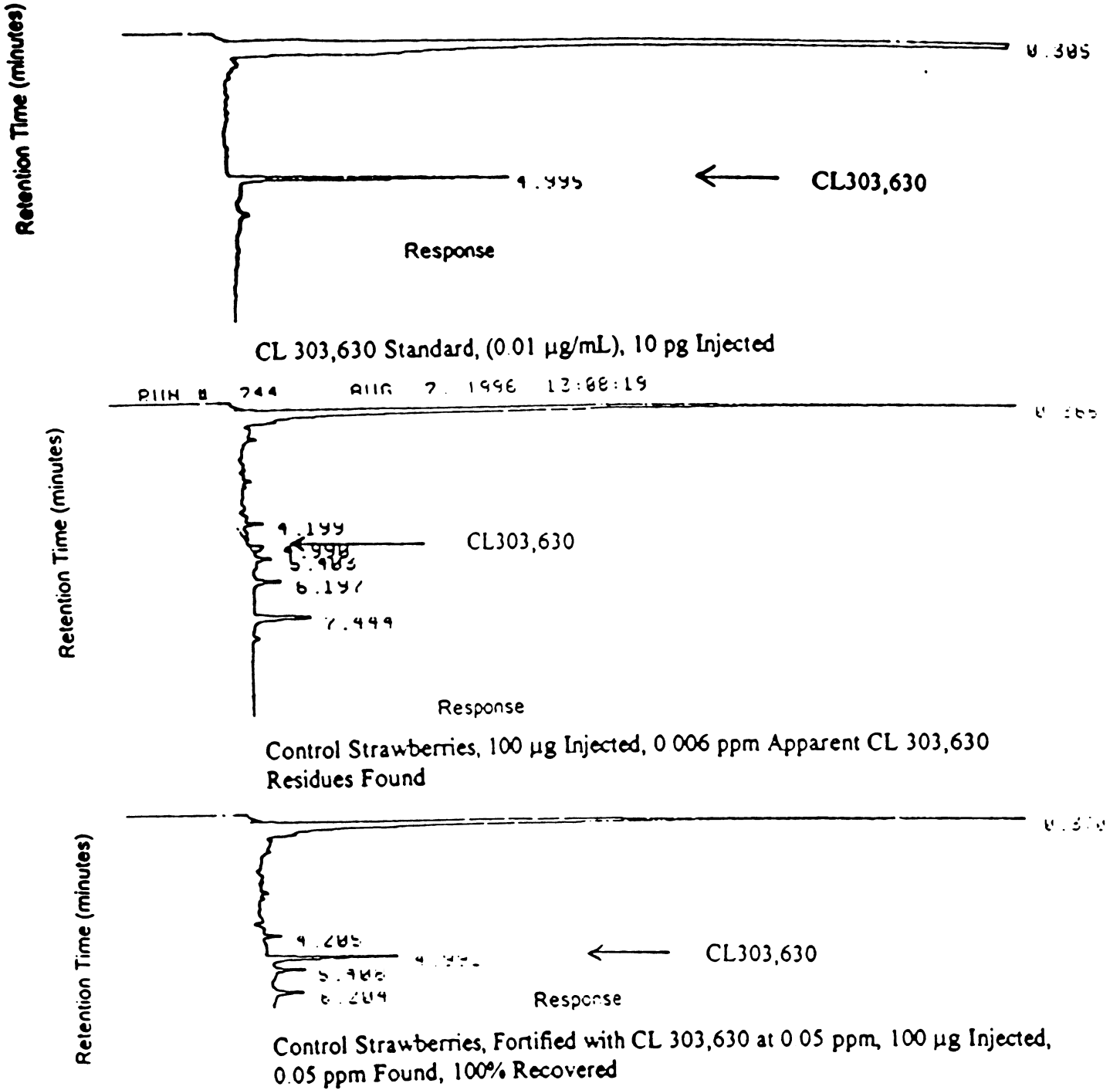


Figure 6: Typical Chromatograms for the Determination of CL 303,630 Residues in White Grapes

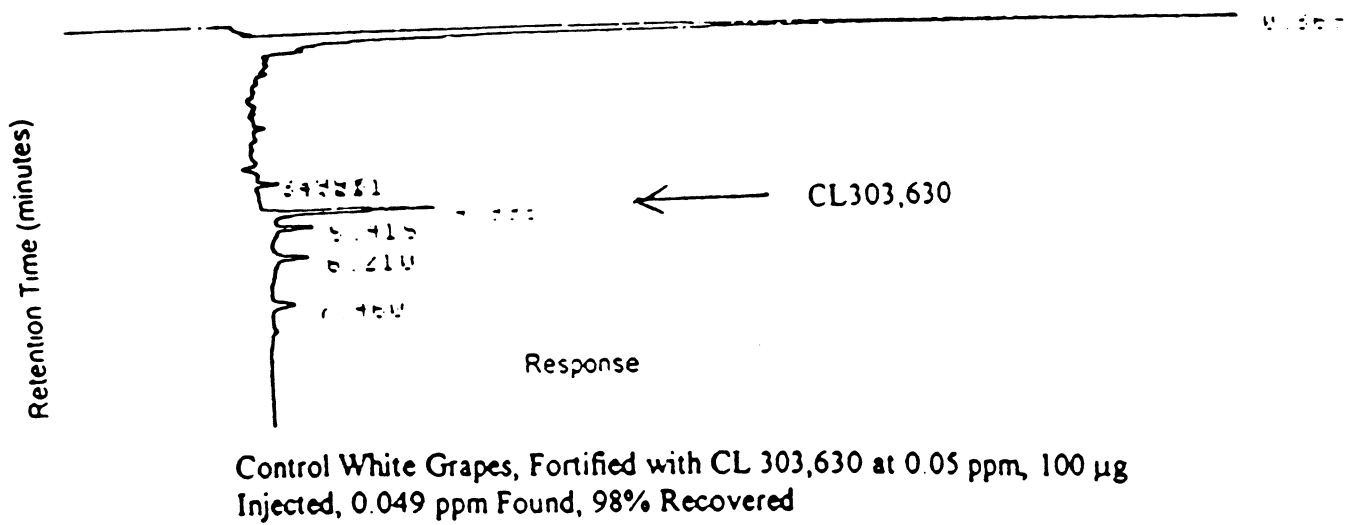
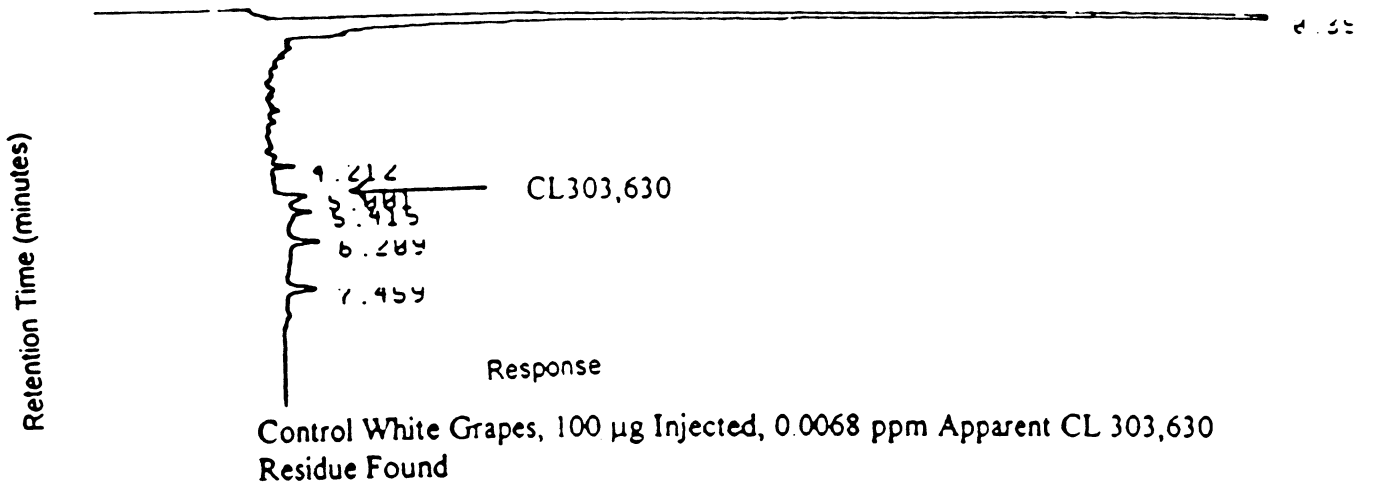
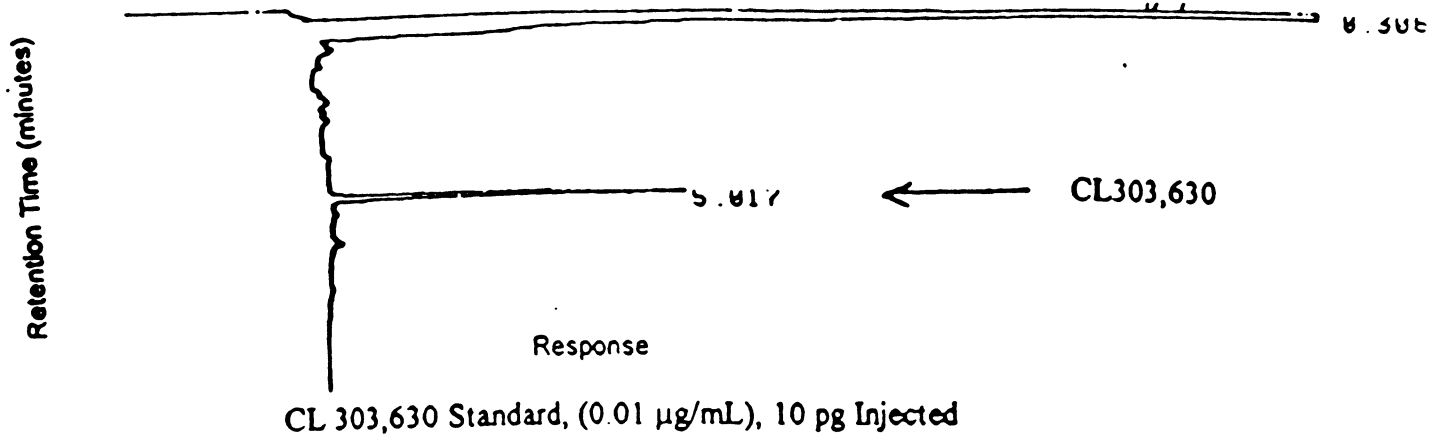


Figure 8: Typical Mass Chromatograms from the Injection of 1 μ L of Working Standard (M.2.)

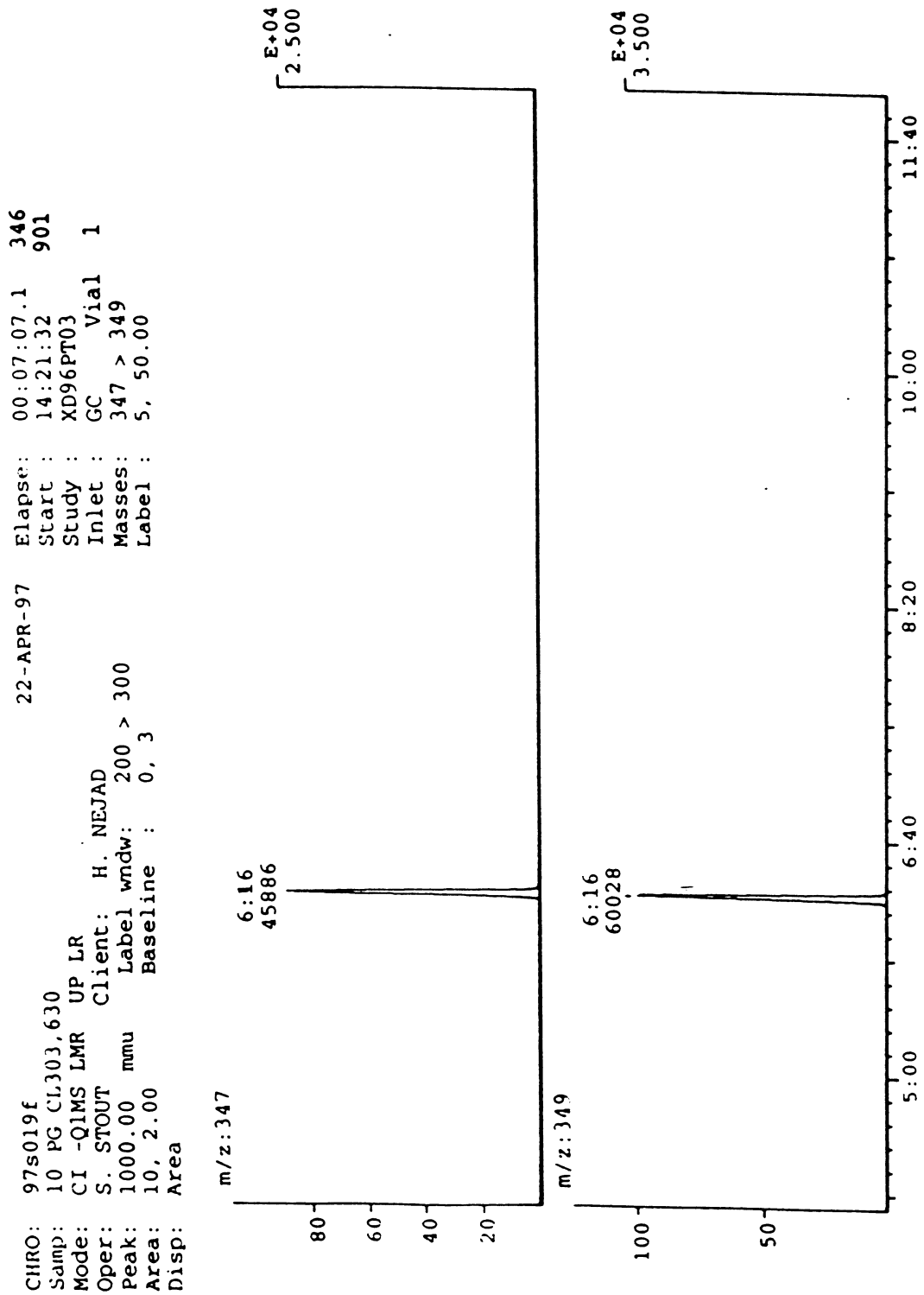


Figure 9: Typical Mass Chromatograms from the Injection of a Control Bing Cherry Sample

Elapse: 00:07:07.5 346
 Start : 14:43:22 900
 Study : XD96PT03
 Inlet : GC Vial 8
 Masses: 347 > 349
 Label : 5, 50.00

22-APR-97
 Client: H. NEJAD
 Label wndw: 200 > 300
 Baseline : 0, 3

CHRO: 97s026a
 Samp: AC10555.46 CONTROL CHERRIES
 Mode: CI -QIMS LMR UP LR
 Oper: S. STOUT
 Peak: 1000.00 mmu
 Area: 10, 2.00
 Disp: Area

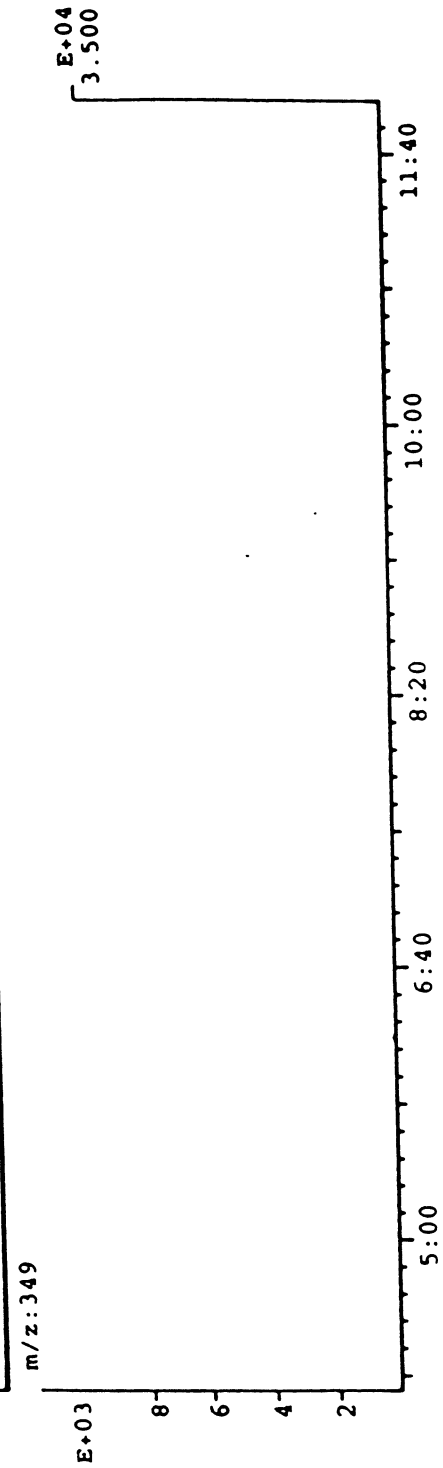
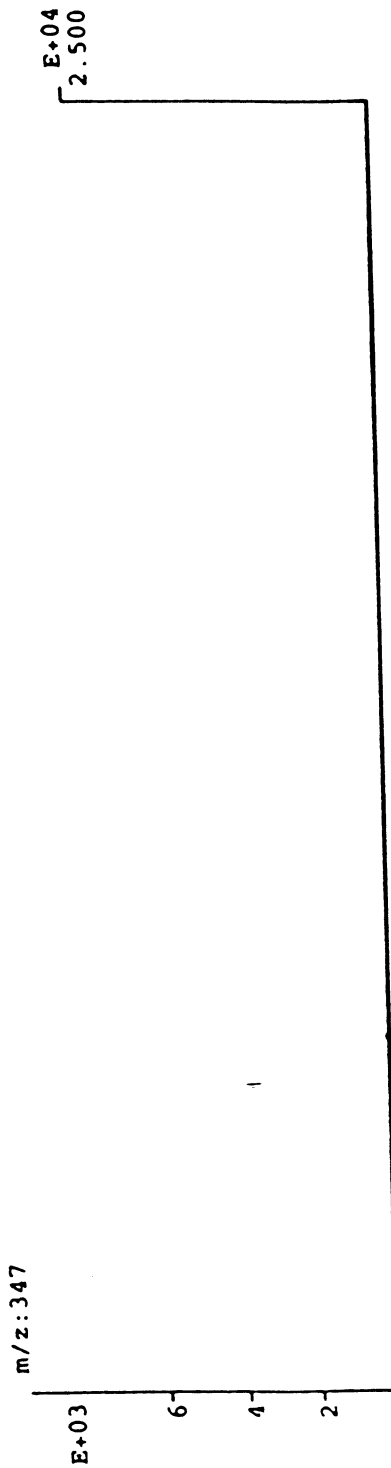


Figure 10: Typical Mass Chromatograms from the Injection of a 0.05 ppm Fortified Bing Cherry Sample

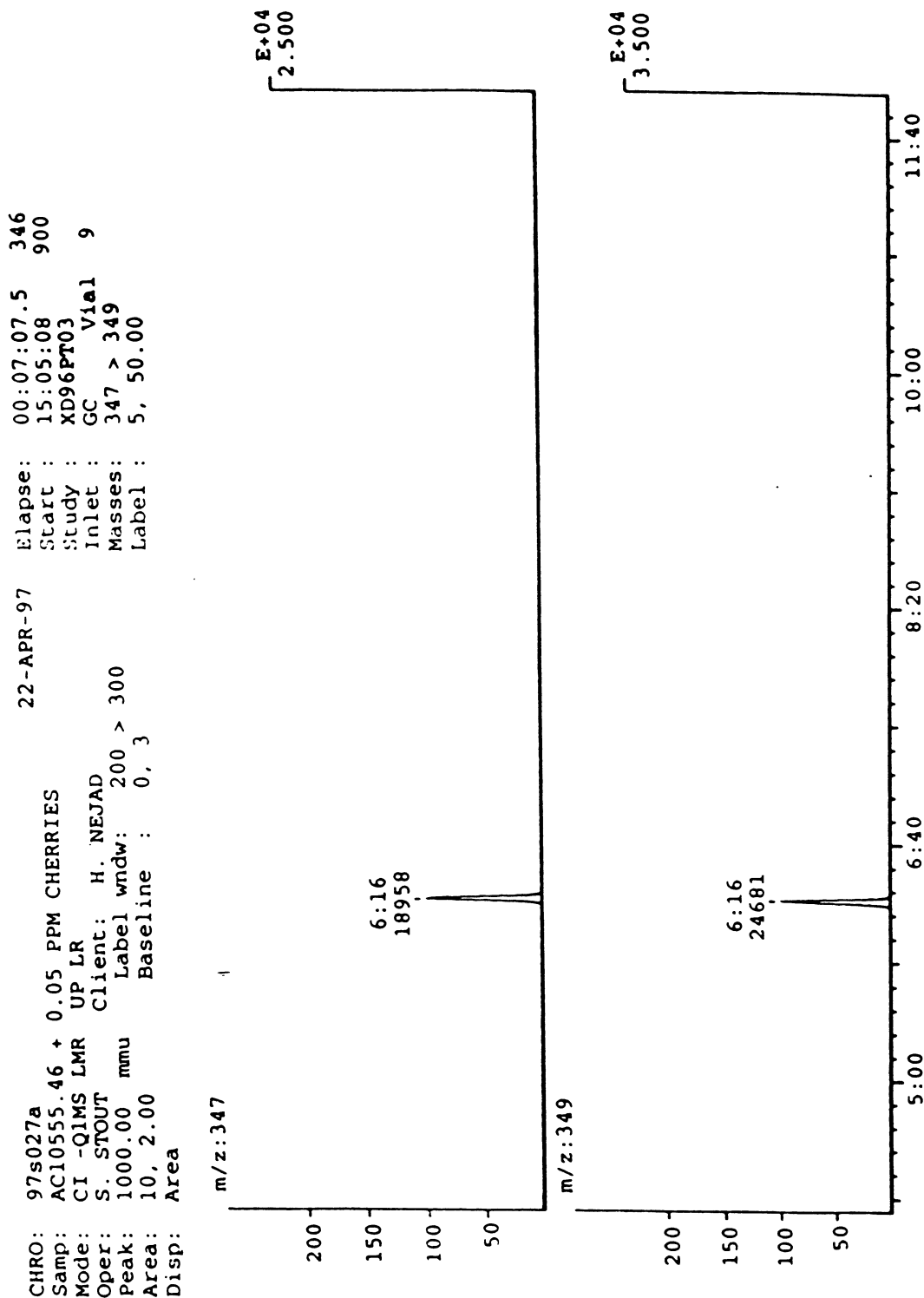


Figure 11: Typical Mass Chromatograms from the Injection of a Control Red Apple Sample

CHRO: 97s020a
 Samp: AC10555.44 CONTROL APPLES
 Mode: CI -QIMS LMR UP LR
 Oper: S. STOUT Client: H. NEJAD
 Peak: 1000.00 mmu Label wndw: 200 > 300
 Area: 10, 2.00 Baseline : 0, 3
 Disp: Area

22-APR-97 Elapse: 00:07:07.5 346
 Start : 11:27:19 900
 Study : XD96PT03
 Inlet : GC Vial 2
 Masses: 347 > 349
 Label : 5, 50.00

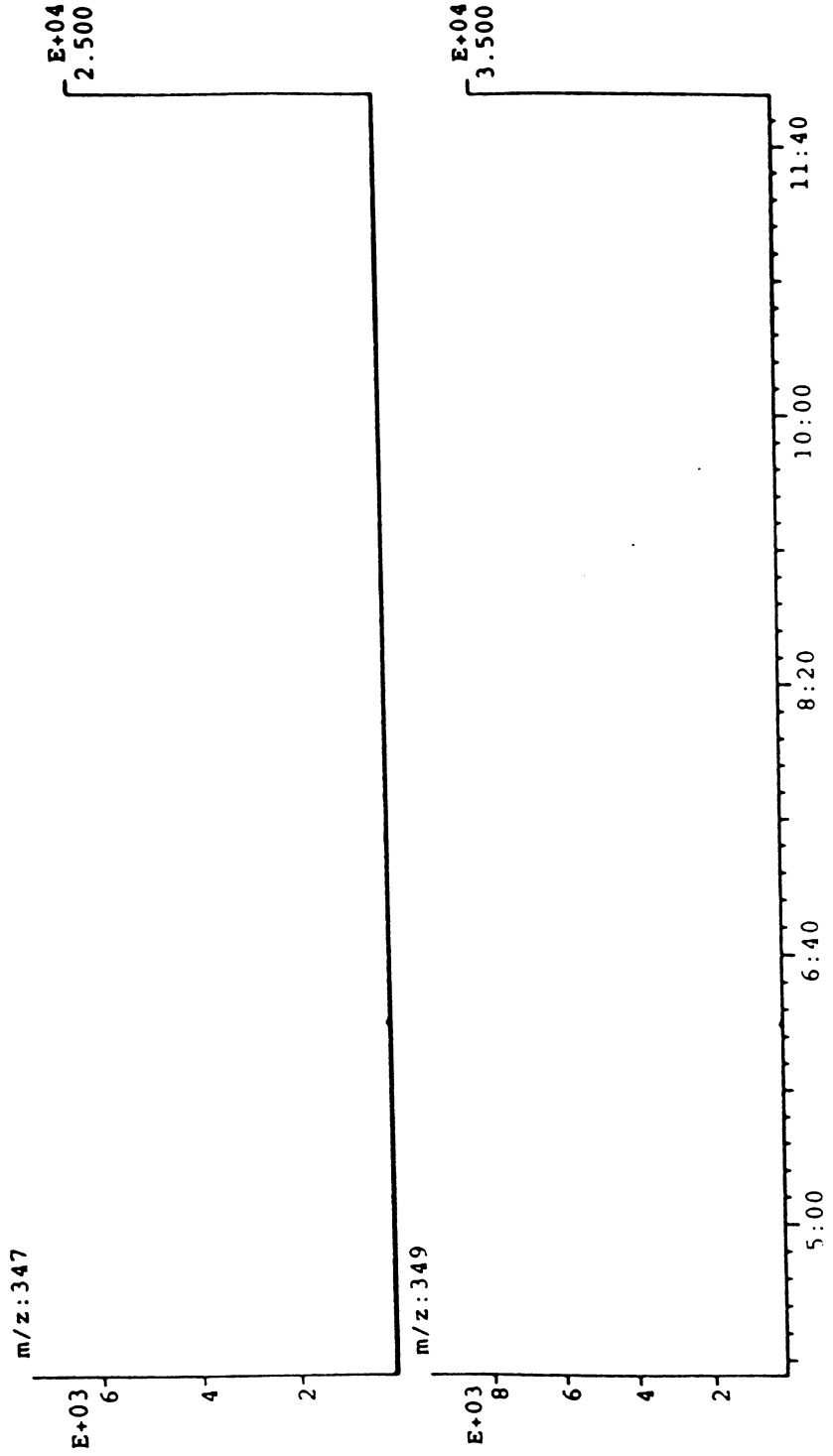


Figure 12: Typical Mass Chromatograms from the Injection of a 0.05 ppm Fortified Red Apple Sample

CHRO: 97s021a
 Samp: AC10555.44 + 0.05 PPM APPLES
 Mode: CI -QIMS LMR
 Oper: S. STOUT
 Peak: 1000.00 mmu
 Area: 10, 2.00
 Disp: Area
 22-APR-97
 Client: H. NEJAD
 Label wndw: 200 > 300
 Baseline : 0, 3
 Elapse: 00:07:07.6 346
 Start : 11:49:02 900
 Study : XD96PT03
 Inlet : GC Vial 3
 Masses: 347 > 349
 Label : 5, 50.00

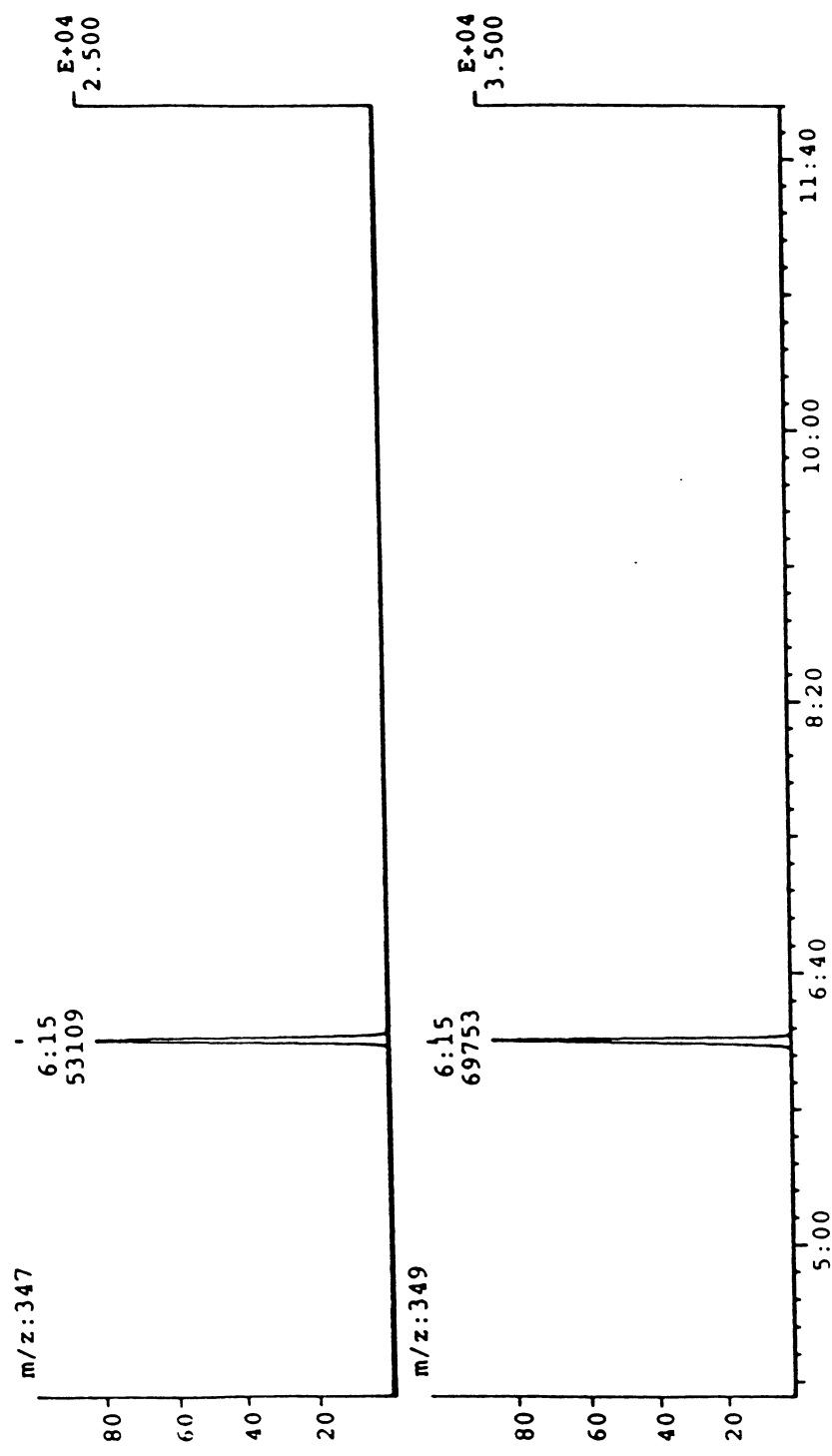


Figure 13: Typical Mass Chromatograms from the Injection of a Control Grape Sample

CHRO: 97s030a
 Samp: AC10555.61 CONTROL GRAPES
 Mode: CI -QIMS LMR UP LR
 Oper: S. STOUT Client: H. NEJAD
 Peak: 1000.00 mmu Label wndw: 200 > 300
 Area: 10.2.00 Baseline : 0, 3
 Disp: Area
 22-APR-97 Elapse: 00:07:07.5 346
 Start : 16:54:09 900
 Study : XD96PT03
 Inlet : GC Vial 12
 Masses: 347 > 349
 Label : 5, 50.00

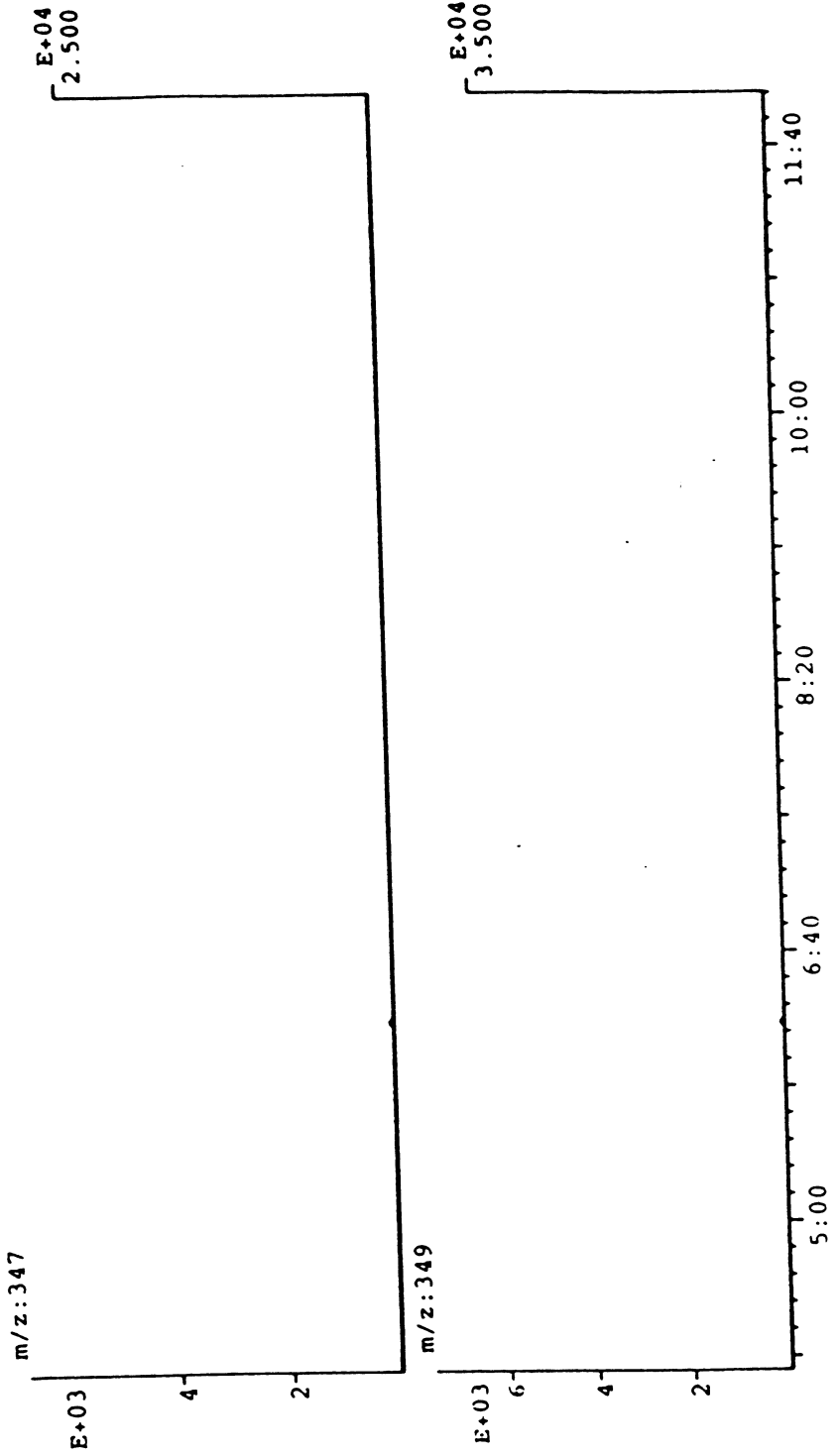


Figure 14: Typical Mass Chromatograms from the Injection of a 0.05 ppm Fortified Grape Sample

CHRO: 97s031a
 Samp: AC10555.61 + 0.05 PPM GRAPES
 Mode: CI -QIMS LMR
 Oper: S. STOUT
 Peak: 1000.00 mmu
 Area: 10, 2.00
 Disp: Area
 Client: H. NEJAD
 Label wndw: 200 > 300
 Baseline : 0, 3
 22-APR-97
 Elapse: 00:07:07.5 346
 Start : 17:15:56 900
 Study : XD96PT03
 Inlet : GC Vial 13
 Masses: 347 > 349
 Label : 5, 50.00

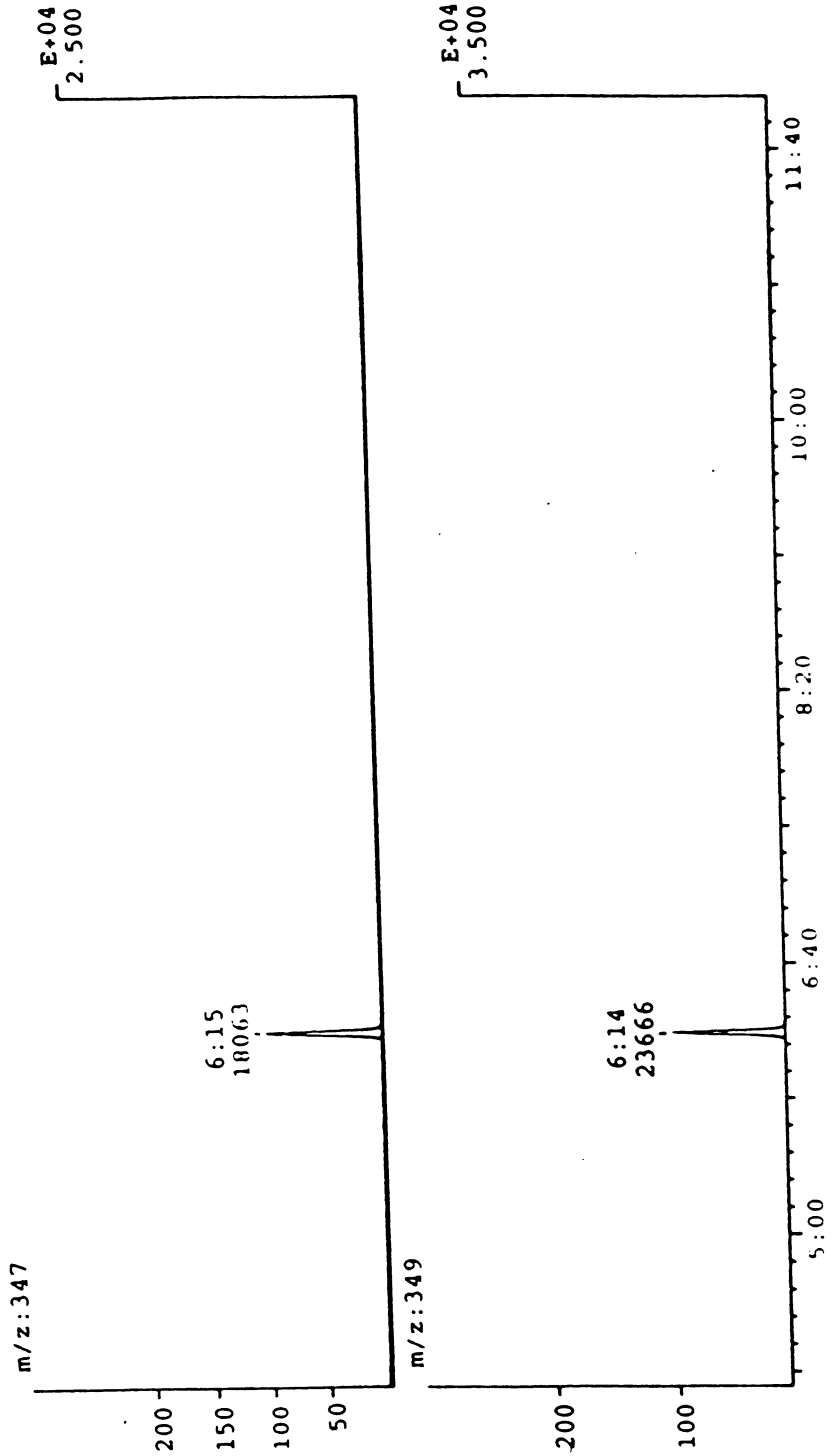


Figure 15: Typical Mass Chromatograms from the Injection of a Control Strawberry Sample

CHRO: 97s032a
 Samp: AC10555.62 CONTROL STRAWBERRIES
 Mode: CI -Q1MS LMR UP LR
 Oper: S. STOUT Client: H. NEJAD
 Peak: 1000.00 mmu Label wmdw: 200 > 300
 Area: 10, 2.00 Baseline : 0, 3
 Disp: Area
 22-APR-97 Elapse: 00:07:07.4 346
 Start : 17:59:32 900
 Study : XD96PT03
 Inlet : GC Vial 14
 Masses: 347 > 349
 Label : 5, 50.00

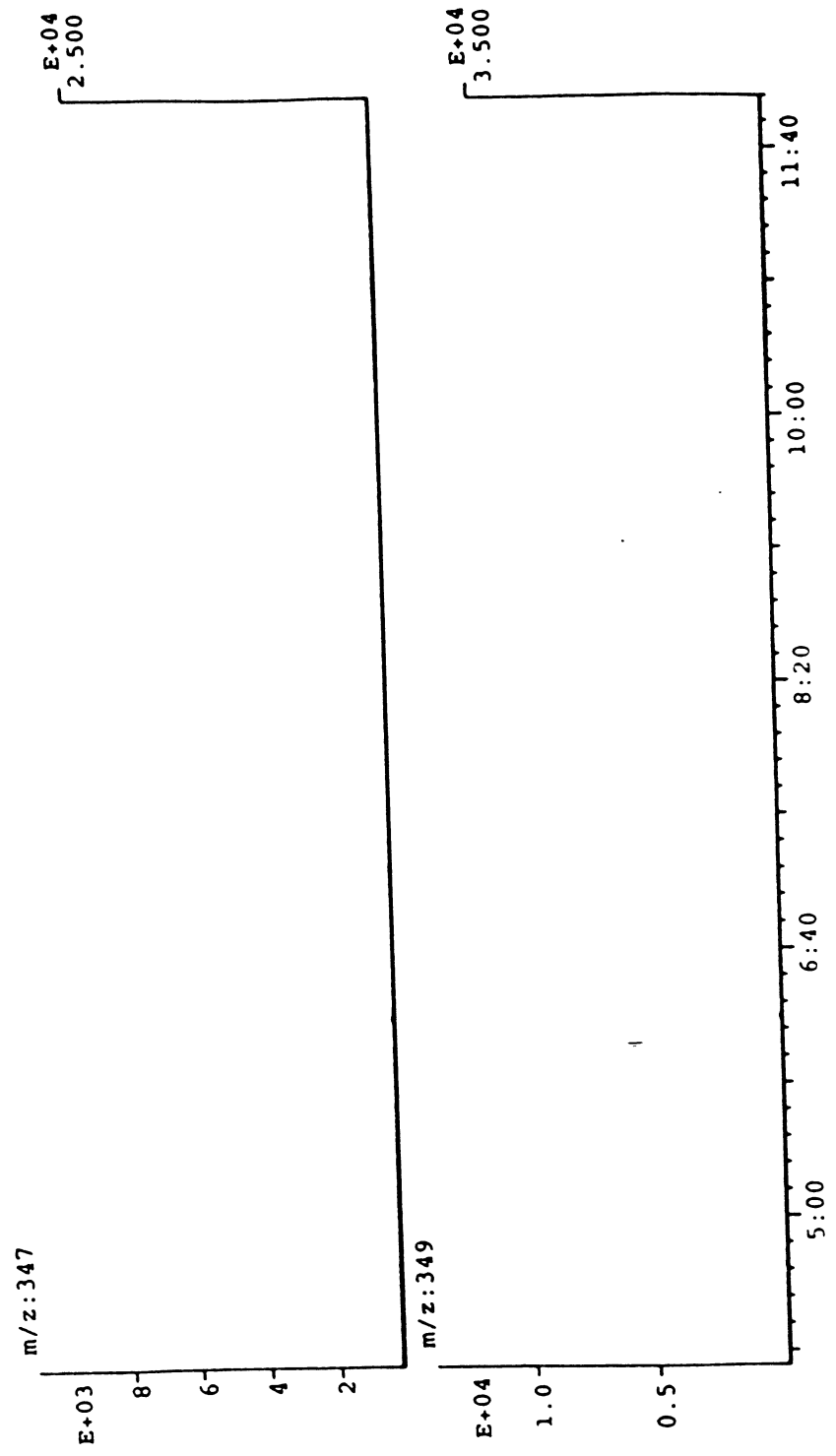


Figure 16: Typical Mass Chromatograms from the Injection of a 0.05ppm Fortified Strawberry Sample

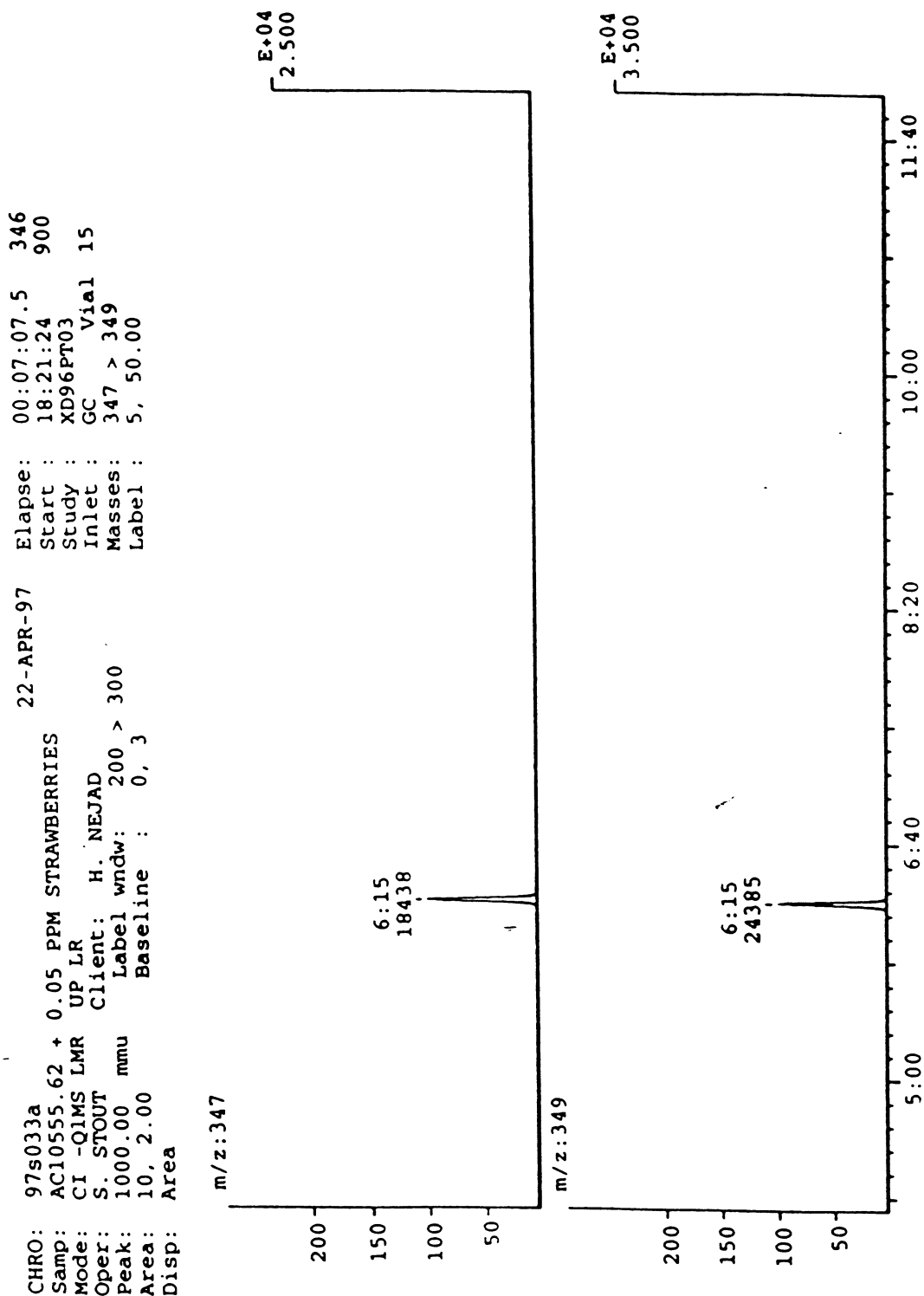


Figure 17: Typical Mass Chromatograms from the Injection of a Control Peach Sample

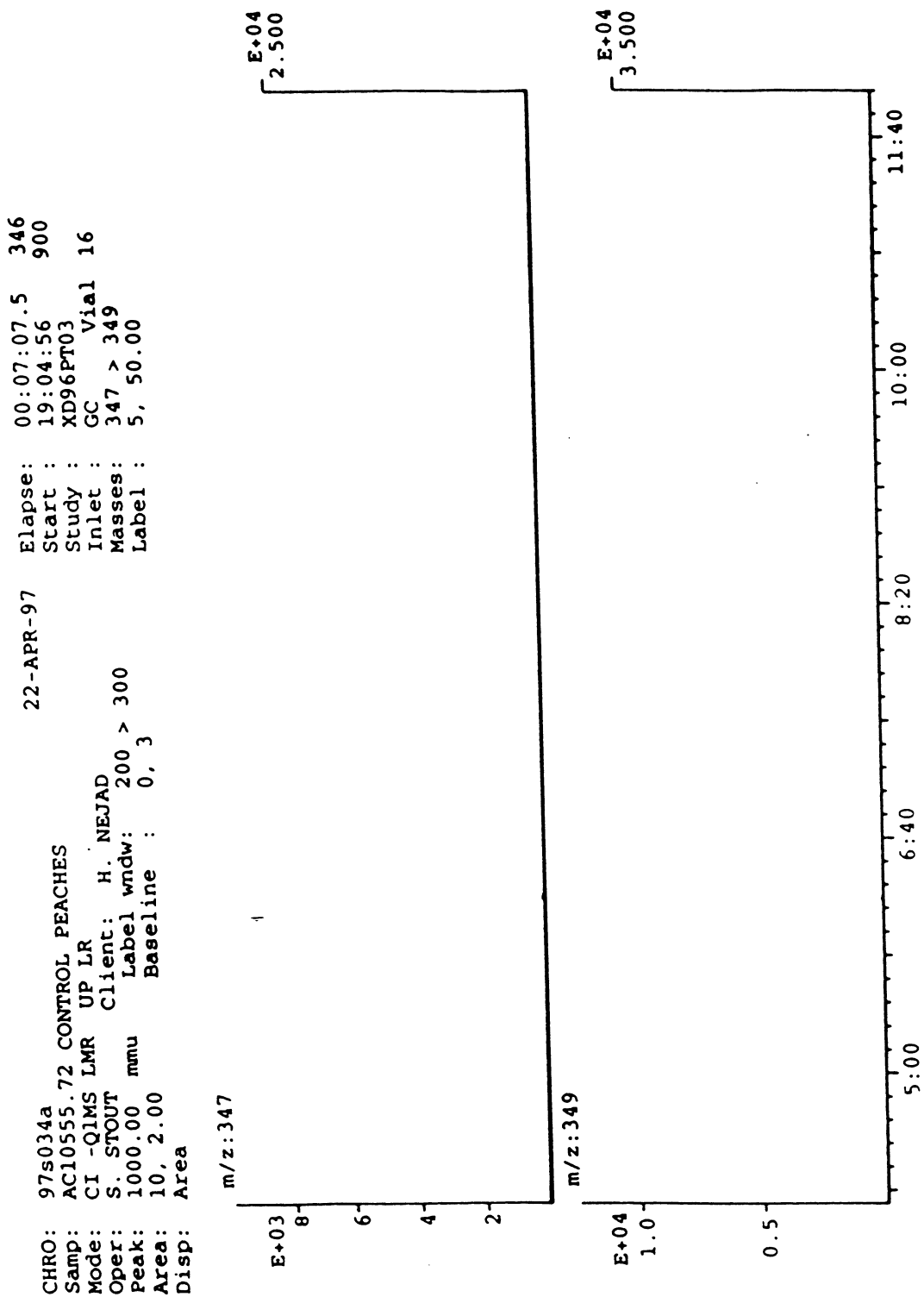


Figure 18: Typical Mass Chromatograms from the Injection of a 0.05 ppm Fortified Peach Sample

