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CGA-152005

ANALYTICAL METHOD TITLE

ANALYTICAL METHOD FOR THE DETERMINATION OF CGA-152005 IN CROPS
BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY WITH COLUMN
SWITCHING INCLUDING VALIDATION DATA
(SUPERSEDES AG-590 ,EPA MRID NO. 43159350)

DATA REQUIREMENT

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AUTHOR

T L OAKES

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PERFORMING LABORATORY

Biochemistry Department
Ciba Crop Protection
Ciba-Geigy Corporation
Greensboro, NC 27419

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ANALYTICAL METHOD NO AG-590C

**Ciba Crop Protection
Ciba-Geigy Corporation
Post Office Box 18300
Greensboro, NC 27419-8300**

VOLUME 1 OF 1 OF STUDY

PAGE NO. 1

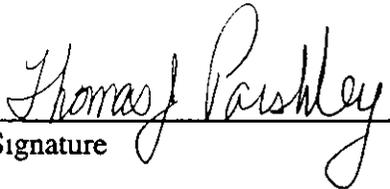
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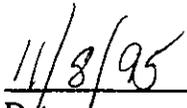
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Company Ciba Crop Protection, Ciba-Geigy Corporation

Company Representative Thomas J. Parshley

Title Senior Regulatory Manager


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STATEMENT CONCERNING GOOD LABORATORY PRACTICES

This volume containing Analytical Method No AG-590C was written to report modifications to the original method to remedy low grain procedural recoveries experienced during a PR-88-5 ruggedness trial, and supersedes Analytical Method No AG-590 (EPA MRID No 43159350)

No new analytical data is reported and the Good Laboratory Practices Compliance Statement (40 CFR Part 160, October 16, 1989) appearing in the original document and signed by the Study Director, applies herein as well

W.T. Beidler
W T Beidler, Ph D , Manager, Residue Chemistry
And Representative of Submitter/Sponsor

11-8-95
Date

SUBMITTER/SPONSOR: Ciba Crop Protection, Ciba-Geigy Corporation, P. O Box 18300, Greensboro, NC 27419-0300

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SUBMITTER/SPONSOR: Ciba Crop Protection, Ciba-Geigy Corporation, Post Office Box 18300, Greensboro, NC 27419

Ciba-Geigy Corporation
Ciba Crop Protection
Biochemistry Department
410 Swing Road
Post Office Box 18300
Greensboro, NC 27419

ANALYTICAL METHOD FOR THE DETERMINATION OF CGA-152005
IN CROPS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY WITH
COLUMN SWITCHING INCLUDING VALIDATION DATA

ANALYTICAL METHOD NO. AG-590C
(Supercedes AG-590, AG-590A and AG-590B)

PROJECT NUMBER: 168982

SUBMITTED BY: T. L. Oakes
D. D. Campbell
S. S. Pyles

TITLE: Chemist IV
Chemist IV
Chemist I

SIGNATURE: *T. L. Oakes*

COMPLETION DATE: *10-3-95*

APPROVED BY: W. T. Beidler

TITLE: Manager, Residue Chemistry

SIGNATURE: *W. T. Beidler*

DATE: *10-3-95*

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I. SUMMARY AND INTRODUCTION

A. SCOPE

Analytical Method AG-590C is a revision of AG-590B¹. AG-590C was written to include modifications to the method to remedy low grain procedural recoveries experienced during a PR-88-5 ruggedness trial². AG-590B clarifies issues resulting from the Petition Method Validation (PMV) trials discussed in Memorandum PP#4F04336 from the United States Environmental Protection Agency. Analytical Method AG-590B also includes a confirmatory method of analysis. Validation information concerning the confirmatory method can be found in Ciba Analytical Method AG-642³. The purpose of AG-590A⁴ was to expand the modifications and potential problems section to include helpful information gathered during a PR 88-5 ruggedness trial⁵. AG-590⁶ contains the results of the method validation study. Analytical Method AG-590C is for determination of residues of CGA-152005 in crops and crop fractions. The limit of detection of this method, determined by the smallest standard amount injected, is 0.8 ng of CGA-152005. The limit of quantitation as determined by fortification experiments is 0.01 ppm. The chemical names and structures of CGA-152005 are shown in Figure 1.

B. PRINCIPLE

A 6-g subsample of crop substrate is homogenized twice with acetonitrile (ACN)/aqueous sodium bicarbonate. Both extracts are filtered through glass wool and combined. A 150-ml aliquot of extract is transferred to a flask and the volume reduced to <0.5 ml. The concentrated extract is diluted with saturated sodium chloride solution and sodium carbonate solution and partitioned against methyl-tert butyl ether (MTBE)/hexane. The aqueous solution is retained and acidified with dilute phosphoric acid before being loaded onto a 20-ml Extrelut cleanup column. The sample on the Extrelut column is partitioned with dichloromethane (DCM)/hexane and the organic solution is collected. The sample solution is evaporated to incipient dryness and the residue reconstituted in ACN/aqueous

ammonium hydroxide. Residue determination is done by narrow bore HPLC with column switching (250 x 2.0 mm Cyano column to a 250 x 2.1 mm Supelcosil LC-18-DB column) with UV detection at 225 nm. See Figure 3 for analytical flowchart.

Oil samples (5-g) are dissolved in 50 ml of hexane and partitioned with carbonate solution. The aqueous layer is diluted with saturated sodium chloride solution and back partitioned with hexane before being acidified and taken to the Extrelut column as above. See Figure 4 for analytical flowchart.

II. MATERIALS AND METHODS

A. APPARATUS

- 1.0 Bottles, square amber wide mouth, 8 oz.
- 2.0 Bottles, Boston Round, narrow mouth, 8 oz.
- 3.0 Filtering crucible holder (Carbon filter tube) (Fisher Cat. #08-261E)
- 4.0 Concentration tube, minimum volume 25-ml
- 5.0 Disposable Pasteur pipets
- 6.0 Erlenmeyer flask, 125-ml, 250-ml
- 7.0 Funnel, long stem, 12.5-cm size
- 8.0 Funnel, powder, 80-mm
- 9.0 Funnel, separatory, 60-ml and 125-ml with Teflon stopcock
- 10.0 Glass wool
- 11.0 Graduated cylinder, 50-ml, 100-ml or equivalent
- 12.0 Homogenizer, Polytron or equivalent
- 13.0 Round bottom flasks, 500-ml, 250-ml
- 14.0 Rotary evaporator, Büchi or equivalent

- 15.0 Syringe filter, ACRODISC LC13 PVDF, 0.2 μm
(Gelman #4450)
- 16.0 Stopcock, 2-way, nylon (ISOLAB, Inc. #QSV)
- 17.0 Syringe, glass multifit 2 and 5-cc size
- 18.0 Vials, Wheaton, 2-ml or equivalent
- 19.0 Volumetric pipets, 1-ml, 2-ml, 8-ml, 10-ml

B. REAGENTS

- 1.0 Acetonitrile (ACN), HPLC grade
- 2.0 Ammonium hydroxide (NH_4OH), ACS Reagent grade
- 3.0 0.05% conc. NH_4OH /water (v/v)
- 4.0 Dichloromethane (DCM), HPLC grade
- 5.0 50% DCM/Hexane (v/v)
- 6.0 Hexane, HPLC grade
- 7.0 Methyl-tert butyl ether (MTBE), HPLC grade
- 8.0 Phosphoric acid (H_3PO_4) conc., Certified ACS grade
- 9.0 0.8% conc. H_3PO_4 /water (v/v)
- 10.0 Sodium chloride, Certified ACS grade
- 11.0 Saturated solution of sodium chloride in water
- 12.0 Sodium bicarbonate, Certified ACS grade
- 13.0 Sodium carbonate, Certified ACS grade
- 14.0 0.4% Sodium carbonate/water (w/v)
- 15.0 8:2 ACN:0.1% Sodium bicarbonate/water (w/v)
- 16.0 Water, HPLC grade

- 17.0 Extrelut® QE, 20-ml capacity (EM Separations cat. #901020-1)
- 18.0 CGA-152005, Analytical Standard supplied by Ciba-Geigy Corporation, 410 Swing Road, Greensboro, NC 27419

C. ANALYTICAL PROCEDURES

1.0 Sample Preparation

Samples are received and stored frozen at -20°C (Ciba SOP 7.20). Samples are prepared under the general guidelines of the US Food and Drug Administration Pesticide Analytical Manual Volume I, Section 141 (Ciba SOP 7.21).

2.0 Extraction

2.1 **OIL SAMPLES:** Transfer 5 g of crude or refined oil to a 125-ml flask. Fortify with CGA-152005 at this point for recovery samples. Add 50 ml of hexane to dissolve the sample. Transfer the organic solution to a 125-ml separatory funnel. Rinse the flask with precisely 10 ml of 0.4% sodium carbonate solution and add this rinse to the separatory funnel. Gently shake the separatory funnel for 3 minutes, then allow the phases to separate (Caution: emulsions form easily). Drain the lower, aqueous phase and any remaining emulsion back into the flask and discard the upper, organic layer.

Add 8 ml of saturated sodium chloride solution to the aqueous solution in the flask and transfer the combined volumes back into the separatory funnel. Add 25 ml of hexane to the separatory funnel and shake for one minute. Allow the layers to separate, then drain the lower, aqueous layer into the 125-ml flask and carry this solution on to Section II.C.4.1. Discard the organic layer.

2.2 CROP RAC'S AND SOLID FRACTIONS:

Weigh a 6-g aliquot of crop substrate into an 8 oz. wide mouth jar. Fortify with CGA-152005 at this point for recovery samples. Immediately add 90 ml 8:2 ACN:0.1% sodium bicarbonate/water and let the sample steep for 15 minutes. Homogenize the sample with a Polytron homogenizer at medium power (approx. 14,000 rpms) for 30 seconds. Filter the sample through a plug of glass wool at the apex and stem of a carbon filter tube into an amber Boston round bottle, or Erlenmeyer flask if for immediate use. Return any crop matrix in the carbon filter tube and the glass wool to the extraction jar. Rinse any matrix residue adhering to the carbon filter tube into the extraction jar with 90 ml 8:2 ACN:0.1% sodium bicarbonate solution.

Homogenize the sample plus glass wool and solvent again for 30 seconds and filter the extract through a new plug of glass wool at the apex and stem of the carbon filter tube. Collect both extracts in the same container and refrigerate the sample extract if it is not to be used immediately.

3.0 Partition Cleanup

3.1 Transfer a 150-ml aliquot of sample extract to a 500-ml round bottom flask and remove the solvent by rotary vacuum evaporation until the volume is <0.5 ml (bath temperature <40°C). Due to the aqueous makeup of this solution evaporation time can vary from 20 minutes to 1 1/2 hours depending on the efficiency of the evaporation system. Evaporation time has not been determined as a factor in loss of CGA-152005 from procedural recovery samples, however, it is recommended to maximize the efficiency of evaporators in order to minimize analysis time. Add 10 ml of

0.4% sodium carbonate solution to the round bottom flask and sonicate to loosen or dissolve any adhering residue. Transfer the solution to a 60-ml separatory funnel (See Section II.H.3.0 for problems with sample solution pH ranges).

- 3.2 Add 8 ml of saturated sodium chloride solution to the 500-ml round bottom flask and swirl. Swirl and sonicate sample to ensure that residues go into solution. Transfer the solution to the 60-ml separatory funnel in Section II.C.3.1. Add 25 ml of 1:1 MTBE:hexane to the 500-ml round bottom flask and swirl. Transfer the solution to the 60-ml separatory funnel above.
- 3.3 Stopper the 60-ml separatory funnel and shake for one minute, taking care to vent the funnel. Allow the two layers to separate. Avoid shaking samples vigorously. A steady rotation of the separatory funnel is effective and allows for adequate partitioning and helps avoid the formation of emulsions. Break any emulsion that may form and drain the lower, aqueous layer and any remaining emulsion back into the 500-ml round bottom flask from Section II.C.3.2. Discard the upper, organic layer and transfer the aqueous layer back to the separatory funnel.
- 3.4 Add 25 ml of 1:1 MTBE:hexane to the 60-ml separatory funnel, stopper and shake for one minute. Allow layers to separate. Break any emulsion that may form and drain the lower, aqueous layer and any remaining emulsion back into the 500-ml round bottom flask from Section II.C.3.3. Discard the upper, organic layer.

4.0 Extrelut Cleanup

4.1 Add aqueous fraction back into 60 ml separatory funnel from Section II.C 3.4. Add 8 ml of 0.8% phosphoric acid to the 500-ml round bottom flask from Section II.C.3.4 (or the flask from Section II.C.2.1 for oil samples) and swirl to mix. Transfer acid to the separatory funnel containing the aqueous fraction, stopper, and mix thoroughly. (See Section II.H.3.0 for problems with sample solution pH ranges.) Transfer the acidic sample solution to the 20-ml Extrelut cleanup column. (See Section II.H.4.0 concerning the preparation of Extrelut columns.) Allow the solution front to migrate to the bottom of the column. Then allow the solution to sit on the Extrelut column for 5-10 minutes to ensure adequate adsorption.

4.2 Rinse the round bottom flask that contained the acidic solution from section II.C.4.1 with 100 ml of 1:1 DCM:hexane. Attach a reservoir to the Extrelut column and partition the sample with the 100 ml of 1:1 DCM:hexane by first passing the DCM:hexane through the 60 ml separatory funnel. The flow through the Extrelut should be no greater than 2-3 ml per minute. The flow may be controlled by attaching a nylon stopcock to the outlet of the column. **NOTE: SIGNIFICANT LOSSES HAVE BEEN NOTED DURING THIS STEP IF ALL GLASSWARE THAT HAS CONTAINED THE ACIDIFIED SOLUTION IS NOT RINSEI AND LOADED ONTO THE COLUMN.** Sonicate the round bottom flask, and stoppering and shaking the flask during this rinse is suggested. Collect the organic solution in a 250-ml round bottom flask. **CAUTION:** If any aqueous solution breaks through the Extrelut column, remove

it by pipet or by running the sample through a plug of absorbent cotton or sodium sulfate before proceeding. Rinse the cotton or sodium sulfate plug with an additional 25 ml of DCM:hexane (1.1). No acidic aqueous solution should be present before evaporation. Evaporate samples to approximately 10 ml on rotovaps (bath temp <35°C). Then remove the samples from the water bath and continue to evaporate just to dryness without any applied heat and reconstitute in the appropriate volume (1.5 mls for screening level) of 20% ACN/0.05% ammonium hydroxide solution. Sonicate and vortex stir the sample before filtering through a 0.2- μ m syringe filter into a vial for analysis by HPLC.

D. INSTRUMENTATION

1.0 Description and Operating Conditions

1.1 Install the HPLC system according to Table I and Figure 2. Control of the switching valve is accomplished via time-programmed contact closures of the detector, autoinjector or other timing source.

1.2 Determine the retention time of CGA-152005 on Column #1 by connecting Column #1 directly to the detector and injecting 24 ng of the analyte. (Inject 40 μ l of the 0.6 ng/ μ l standard solution prepared in Section II.I.1.0.) A typical window of approximately 2-3 minutes should be used. Time the cut approximately 30 seconds before and after the analyte peak. Column 1 should be reprofiled whenever new mobile phase is prepared or when detector response of standards vary more than 10% from previous runs. Occasionally when profiling column #1, a split peak occurs. This seems to be caused by a titration effect between the basic

sample solution and the acidic mobile phase. If this occurs, cut approximately 30 seconds before the first peak and 30 seconds after the second peak to ensure transfer of the entire peak. The peak will refocus into a single peak as it enters the analytical column.

- 1.3 Reconnect the system as shown in Figure 2. Program the valve to switch to the INJECT POSITION at the beginning of the CGA-152005 analyte peak and to return to the LOAD POSITION at the end of the analyte peak of CGA-152005, as determined in Section II.D.1.2.
- 1.4 Inject 24 ng of CGA-152005 to determine its retention time through the two columns and to confirm that the valve time programming is correct.

2.0 Standardization

- 2.1 Calibrate the HPLC system with each analytical run by checking the retention time and detector response relative to previous runs. Retention times must not vary more than 2% within a run and detector response should not vary more than 10% between runs.
- 2.2 Standardize the HPLC system by injecting 40- μ l aliquots of standard solutions of CGA-152005 in a working range of 0.8-24 ng/injection (Figure 5). Generate a linear regression from the data by comparing detector response and ng injected (Table II).

E. INTERFERENCES

None.

F. CONFIRMATORY TECHNIQUES

A triple column switching confirmatory system has been developed and validation data can be found in Ciba Analytical Method AG-642. This system incorporates a phenyl column as the analytical column, and the original analytical column (Supelcosil LC-18-DB) is converted to a second preparatory column. This is accomplished by adding a second switching valve and a third HPLC pump. (See Table Ia and Figure 2a for details and operating condition)

G. TIME REQUIRED

A skilled analyst can complete the extraction and analysis of a set of 6-8 samples in 10-12 working hours.

H. MODIFICATIONS AND POTENTIAL PROBLEMS

- 1.0 Some samples may develop emulsions after shaking (Section II.C.3.3 and II.C.3.4). These may be cleared if allowed to settle out slightly and then gently stirred with a glass rod. Centrifugation can also be used to settle emulsions. It is important that the organic layer be clear of emulsion before separation. Grain samples are especially subject to loss of analyte in the uncleared organic layer during partitions. In addition, any small amounts of remaining emulsion should be taken forward through the procedures.
- 2.0 After fortification, samples should not stand at room temperature for a prolonged period of time before extraction.
- 3.0 For most samples, the pH of the aqueous solutions will be in the optimum range during the cleanup procedures. However, an occasional sample may be more acidic or basic than average, and this can lead to loss of analyte. It may be necessary to check the solution pH of problematic samples at two places. In Section II.C.3.1 the pH of the sample solution should be 11 ± 1 after addition of the carbonate solution. In Section II.C.4.1,

the pH of the sample solution should be 3.0 + 1 after addition of dilute phosphoric acid. If sample solutions fall outside the suggested pH range, then concentrated phosphoric acid or sodium hydroxide should be used for correction.

NOTE: WHEN TRYING TO OBTAIN AN ACIDIC PH OF 3.0, IF SOLUTION IS CLOSE, THE ADDITION OF 1 EXTRA ML OF THE 0.8% PHOSPHORIC ACID SOLUTION SHOULD BE ADEQUATE TO ACHIEVE THIS PH.

- 4.0 When preparing the Extrelut columns, the columns should be inverted and tapped gently several times in order to remove any fine particles of the diatomaceous earth that may exist. These small particles could cause columns to drip slowly or even clog. The frit at the top of the column should then be pressed against the top of the column bed. Note: Lot Numbers of Extreluts can be checked for suitability by running a control or reagent blank through the method up to Section 4.0. At this point a known amount of standard solution can be added and run through the Extrelut in order to test the efficiency of the Extreluts.
- 5.0 During the evaporation of sample solutions in Section II.C.4.2, any water bath used must not have a temperature >35°C and the samples should be removed as soon as they are ready. Excessive temperature, especially when the sample has gone to dryness, leads to analyte decomposition. The final evaporation to dryness must be done without external heating (during validation of this method, a vacuum centrifuge evaporator was used without applied heat, which kept the samples cold during evaporation).
- 6.0 Stopping Points: Refrigerated extracts have shown stability for up to 72 hours. Extract aliquots can also be evaporated to about 20-ml for overnight refrigerated storage. The DCM/hexane partition eluate may be stored refrigerated overnight prior to any evaporation.

I. PREPARATION OF STANDARD SOLUTIONS

1.0 Preparation of Standard CGA-152005 Solutions

- 1.1 Weigh 10 mg of CGA-152005 analytical standard into a 100-ml volumetric flask and dilute to the mark with ACN.
- 1.2 Make serial dilutions of the 0.1 mg/ml standard solution with 20% ACN/0.05% ammonium hydroxide solution (w/v) to give a series of fortification/analytical standards in a range of 0.02 µg/ml to 3.0 µg/ml of CGA-152005. Store the standard solutions in amber bottles at 4°C in the dark when not in use. Standards have been successfully used for up to four months after preparation.
- 1.3 CGA-152005 is degraded in methanol. No solubility problems have been observed with CGA-152005 in the solvents used.

J. METHODS OF CALCULATIONS

1.0 Determination of Sample Residues

- 1.1 Inject 40-µl aliquots of sample extracts from Section II.C.4.2. into the HPLC under the same conditions as for standards. Make appropriate dilutions of the samples in 2:8 ACN:0.05% ammonium hydroxide/water solution to bring the sample peak heights within the range of the standard curve. Compare the peak heights of the unknown samples to the standard curve or enter the peak height into a least squares program to determine the nanograms of CGA-152005 in the injected aliquot. Typical chromatograms for control and procedural recovery samples are shown in Figures 6-10.

- 1.2 To calculate the residue results, the mg injected must first be calculated as follows: (Equation 2)

$$(2) \text{ mg inj} = \frac{(G) (V_a) (V_i)}{[V_e + W(M/100)] (V_f)}$$

G = milligrams sample extracted
V_a = aliquot volume
V_e = extraction volume
V_i = injection volume (μl)
V_f = total volume of final injection solution (μl)
R% = recovery ratio given by equation 4
W = grams samples extracted
M = % moisture of substrate

Calculate the residue results in terms of ppm of CGA-152005 by using the following equation (R is expressed as the decimal of the percent value and is not used in calculations for tolerance enforcement purposes.):

$$(1) \text{ ppm} = \frac{(\text{ng CGA-152005 found})}{(\text{mg sample injected})} (R)$$

2.0 Fortification Experiments

This method is validated for each set of samples analyzed by including an untreated control sample and one or more control samples fortified immediately prior to extraction with CGA-152005.

2.1 Add 1.0 ml of a 0.06 μg/ml standard solution of CGA-152005 to 6 g of control crop prior to the addition of extraction solvent for a 0.01 ppm fortification. Use correspondingly larger amounts of standards (volume should not exceed 2 ml) for higher fortifications. Analyze control and freshly fortified samples along with the treated samples according to the procedures of the method.

2.2 Calculate the final ppm value of the control and fortified samples according to the following equation.

$$(3) \text{ ppm CGA-152005} = \frac{\text{ng CGA-152005 found}}{\text{mg sample injected}}$$

Determine the recovery factor by first subtracting the background detector response, if any, in the control sample from the CGA-152005 response in the recovery sample. Calculate the recovery factor as a percentage (R%) by the equation:

$$(4) R\% = \frac{\text{ppm CGA-152005 found}}{\text{ppm CGA-152005 added}} \times 100\%$$

III. RESULTS AND DISCUSSION

Recovery results for fortified control samples were used to calculate accuracy in terms of a mean, standard deviation (sd) and Coefficient of Variation (CV) for the limit of determination, and for all recovery results included in the validation.

The average recovery for samples fortified with CGA-152005 at the limit of determination of 0.01 ppm was 87% (sd: 15, CV: 17%, n=21) and 88% (sd: 13, CV: 15%, n=62) for all levels (See Table III).

Samples from two Metabolism studies^{7,8} were analyzed during method validation. Corn from these studies was treated with either ¹⁴C-phenyl-CGA-152005 or ¹⁴C-triazine-CGA-152005 via stem-injection (greenhouse grown plants) or a 40-g a.i./ha foliar spray (field grown plants).

Precision of the method was determined by calculating the mean, Coefficient of Variation and standard deviation of replicate analysis sets of each of the incurred ¹⁴C-residue samples. Only some of the samples contained both enough plant material for triplicate analysis and ¹⁴C levels high enough to quantitate by LSC and/or HPLC. The results are as follows: Phenyl-¹⁴C-CGA-152005 injected corn foliage, mean = 0.031 ppm, sd: 0.003, CV: 9% (HPLC); phenyl-¹⁴C-CGA-152005 injected corn stalk mean = 0.007, sd: 0.001, CV: 14% (LSC); triazine-¹⁴C-CGA-152005 injected corn foliage, mean = 0.16, sd: 0.04, CV: 25% (HPLC). Overall, the precision of Analytical Method AG-590A is acceptable.

determined by comparing the total ppm ¹⁴C-residue found in the sample from combustion analysis to the ppm ¹⁴C-residue found in the initial sample extract from Section II C.2.0. The formula for the determination of % extractability is:

$$\% \text{ ext.} = \frac{\text{ppm } ^{14}\text{C-residue extract}}{\text{ppm } ^{14}\text{C-residue sample}} \times 100\%$$

The extractabilities for greenhouse grown stem-injected corn substrates were 69% and 102% for grain and foliage/stalk, respectively. The extractabilities for field grown spray-treated corn substrates were 95% and 42% for forages and fodder, respectively. Grain from field treated corn contained total incurred ¹⁴C residues too low to quantitate.

The accountability of an Analytical Method is determined by comparing the total ppm ¹⁴C-residue found in the sample, the ppm ¹⁴C-residue found in the final fraction and the ppm analyte found in the final fraction to each other. The determinations of CGA-152005 by HPLC and of ¹⁴C by LSC in the final fraction solutions correlated very well and showed that the cleanup procedures isolate CGA-152005 from any other metabolites or degradates. Also this Analytical Method was able to extract weathered residues from and determine parent compound in ¹⁴C-CGA-152005 treated samples (Table IV and Figures 11-13).

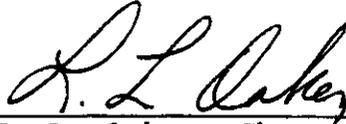
This method has been validated under Protocol No. 106-91 with Analytical Method AG-590A as the final report. Results of this validation are shown in Tables III and IV and are reported in Residue Test Report RI-MV-003-91, No. 1⁹.

IV. CONCLUSION

Analytical Method AG-590C is a valid and accurate method for the determination of parent residues of CGA-152005 in crops.

V. CERTIFICATION

The reports and experimental results included in this study, Laboratory Project I.D. AG-590C, are certified to be authentic accounts of the experiments.



T. L. Oakes, Chemist IV
Residue Chemistry
Biochemistry Department
910-632-2393

10-3-95
Date

TABLE I. LIQUID CHROMATOGRAPHIC OPERATING CONDITIONS FOR ANALYSIS OF CGA-152005

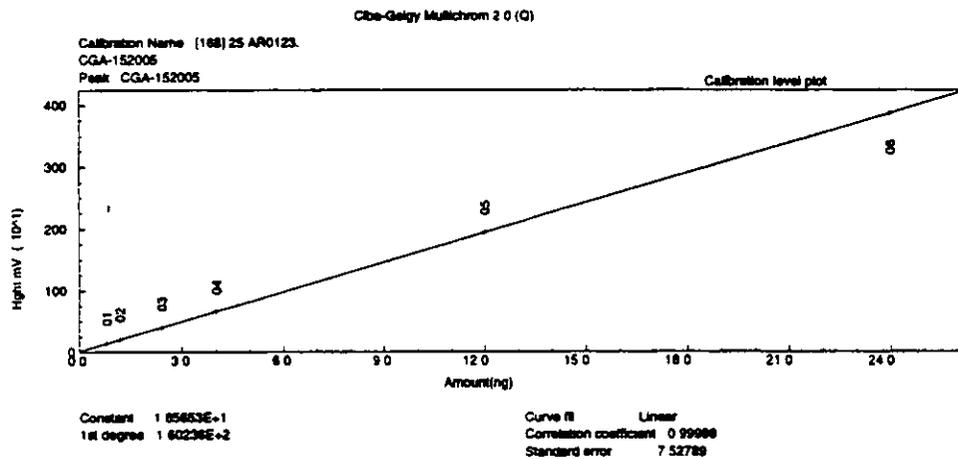
Instrument	Waters 501 HPLC pump (pump 2) or equivalent Perkin-Elmer Model Series-4 Solvent Delivery System (pump 1) or equivalent Perkin-Elmer Model ISS-100 Automatic HPLC sampler or equivalent ABI Spectroflow Model 783 Variable Wavelength UV Detector or equivalent Valco 6-port nitronic valve with electronic actuator or equivalent
Column Oven.	BioRad HPLC column heater, model number 125-0425 or equivalent
Oven Temp .	30°C (both columns)
Column 1.	Brownlee Guard Cartridge, Sphere-5 cyanopropyl, 3 cm X 2 1 mm (Rainin cat. #CS-032) YMC 120A CN, 250 mm x 2 0 mm, 5-µm particle size (YMC Inc. cat #MC-512)
Column 2.	Supelcosil LC-18-DB, 250mm x 2 1 mm, 5-µm particle size (Supelco cat. #5-7940M)
Mobile Phase 1	3.7 ACN:0.1% H ₃ PO ₄ /water
Mobile Phase 2	4.6 ACN:0.1% H ₃ PO ₄ /water
Retention Time:	~14 min. (Column 1) ~30 min (through both columns)
Detection:	ABI Kratos Spectroflow Model 783 Programmable Absorbance Detector or equivalent variable wavelength detector.
Wavelength.	225 nm
Attenuation:	0.006 AUFS
Flow Rate:	0.3-0.4 ml/min (both pumps)
Volume Injected:	40 µl
Run Time:	40 min/injection

TABLE Ia. LIQUID CHROMATOGRAPHIC OPERATING PARAMETERS FOR
THE CONFIRMATORY ANALYSIS OF CGA-152005 BY
AG-642 USING TRIPLE COLUMN SWITCHING

HPLC Pumps	(3) Perkin-Elmer LC250 HPLC pumps or equivalent
Autosampler.	Perkin-Elmer Model ISS-100 Automatic HPLC sampler or equivalent
Column Switching Valves	(2) VICI EQ60 HPLC Switching Valve or equivalent
Column Oven.	BioRad HPLC column heater, model number 125-0425 or equivalent, 30-35°C
Preparatory Column 1	YMC Inc. Cyano, 2 mm X 250 mm, YMC Part No. MC-512
Prep 1 Mobile Phase:	70% 0.1% H ₃ PO ₄ 30% acetonitrile
Flow:	0.3 mL/minute
Typical Retention Time (Tr) on Column 1.	18 minutes
Preparatory Column 2.	Supelco Supelcosil LC18DB, 2.1 mm X 250 mm, Supelco Part No 5-7940
Prep. 2 Mobile Phase:	60% 0.1% H ₃ PO ₄ :40% acetonitrile
Flow.	0.3 mL/minute
Typical Tr through Column 1 and Column 2:	38 minutes
Analytical Column.	YMC Inc. Phenyl, 2 mm X 250 mm, YMC Part No. MC-412
Analyt. Mobile Phase.	50% 0.1% H ₃ PO ₄ 50% acetonitrile
Flow Rate.	0.3 mL/minute
Detection:	UV detection at 225 nm using an ABI Kratos Spectroflow model 783 Programmable Absorbance Detector or equivalent.
Injection Volume:	50 µL
Approximate Tr. through Column 1, Column 2 and the Analytical Column.	48 minutes
Run Time	60 minutes

TABLE II. TYPICAL STANDARDIZATION DATA FOR CGA-152005

Std Wt. Inj. ng	Cal. Lev.	Peak Height mV
0.8000	01	152
1.2000	02	213
2.4000	03	390
4.0000	04	664
12.0000	05	1944
24.0000	06	3863



Reported on 3-AUG-1995 at 10:44

TABLE III. SUMMARY OF RECOVERY DATA FOR CROP SAMPLES FORTIFIED WITH CGA-152005

Sample Number	Corn Substrate	Fortification Level (ppm)	% Recovery
G 00A	Grain	0 (Control)	(<0.01 ppm)
G 01A, G 01B	Grain	0.01	63, 92
G 05A, G 05B	Grain	0.05	73, 76
G 00AR	Grain	0 (Control)	(<0.01 ppm)
G 01AR, G 01BR	Grain	0.01	120, 101
G 05AR, G.05BR	Grain	0.05	86, 69
G 00BR	Grain	0 (Control)	(<0.01 ppm)
G.10AR, G.10BR	Grain	0.10	106, 100
G 20AR, G.20BR	Grain	0.20	97, 98
GT 0C	Grain	0 (Control)	(<0.01 ppm)
GT 01	Grain	0.01	75
GT 02	Grain	0.05	84
FLP 0C, FLT.0C	0-Day Forage	0 (Control)	(<0.01 ppm) (<0.01 ppm)
FLP.10, FLT 10	0-Day Forage	0.1	95, 102
FLP2.0, FLT2 0	0-Day Forage	2.0	89, 97
FLP4 0, FLT4.0	0-Day Forage	4.0	102, 83
XFP 0C	Foliage	0 (Control)	(<0.01 ppm)
FP 01	Foliage	0.01	87
FP 20	Foliage	0.20	89
XFT 0C	Foliage	0 (Control)	(<0.01 ppm)
FT.02	Foliage	0.02	85
FT1.0	Foliage	1.0	83
F 00A	Forage	0 (Control)	(<0.01 ppm)
F.01A, F.01B	Forage	0.01	80, 83
F.05A, F 05B	Forage	0.05	92, 90
F.00B	Forage	0 (Control)	(<0.01 ppm)
F 10A, F 10B	Forage	0.10	73, 72
F 20A, F 20B	Forage	0.20	92, 60
FFP.0C	Forage	0 (Control)	(<0.01 ppm)
FFP 01	Forage	0.01	61
FFP 10	Forage	0.10	101
FFT 0C	Forage	0 (Control)	(<0.01 ppm)
FFT.01	Forage	0.01	110
FFT.05	Forage	0.05	94
FSP.0C, FST.0C	Silage Stage Forage	0 (Control)	(<0.01 ppm) (<0.01 ppm)
FSP.01, FST.01	Silage Stage Forage	0.01	102, 72
FSP.05, FST.05	Silage Stage Forage	0.05	83, 104
SP.0C	Stalk	0 (Control)	(<0.01 ppm)
SP.01	Stalk	0.01	77
SP.10	Stalk	0.20	91
ST.0C	Stalk	0 (Control)	(<0.01 ppm)
ST.01	Stalk	0.01	87
ST.20	Stalk	0.20	80

Mean = 88%, sd = 13, CV: 15%, n=62

* Samples analyzed but rejected due to documented problems during workup or analysis.

TABLE III. SUMMARY OF RECOVERY DATA FOR CROP SAMPLES FORTIFIED WITH CGA-152005 (Continued)

<u>Sample Number</u>	<u>Corn Substrate</u>	<u>Fortification Level (ppm)</u>	<u>% Recovery</u>
D 00A	Fodder	0 (Control)	(<0.01 ppm)
D 01A, D.01B	Fodder	0.01	79, 103
D 05A, D.05B	Fodder	0.05	91, 96
D 00B	Fodder	0 (Control)	(<0.01 ppm)
D 10A, D 10B	Fodder	0.10	68, 99
D.20A, D.20B	Fodder	0.20	72, 112
FDP 0C, FDT.0C	Fodder	0 (Control)	(<0.01 ppm) (<0.01 ppm)
FDP 01, FDT 01	Fodder	0.01	78, 75
FDP 05, FDT 05	Fodder	0.05	72, Rej *
OIL 0	Crude Oil	0 (Control)	(<0.01 ppm)
OIL 01A, OIL.01B	Crude Oil	0.01	Rej.*, 87
OIL 05A, OIL.05B	Crude Oil	0.05	84, 86
FLR.0	Flour	0 (Control)	(<0.01 ppm)
FLR 01A, FLR.01B	Flour	0.01	97, 92
FLR 05	Flour	0.05	102
FLR 10	Flour	0.10	85

Mean = 88%, sd = 13, CV. 15%, n = 62

* Samples analyzed but rejected due to documented problems during workup or analysis.

TABLE IV. SUMMARY OF RESULTS FOR ¹⁴C-CGA-152005 TREATED CORN

Sample ID	Study Number 54-91.1 Code No.	Incurred ¹⁴ C Level (ppm) *	(HPLC) ppm Found	% ¹⁴ C Extracted	ppm ¹⁴ C Found in Final Volume
(Sprayed Phenyl- ¹⁴ C-CGA-152005)					
(0-Day Forage)					
FLP SB	53434	3 44	1.63	94	1.61
(30-Day Forage)					
FFP SA	53435	0.092	<0.01	97	0 002
FFP SB	53435	"	<0.01	92	0 003
FFP.SC	53435	"	<0.01	96	0 002
(46-Day Silage Stage Forage)					
FSP SA	53436	0 034	<0 01	112	<0 001
FSP SB	53436	"	-NA-**	100	-NA-**
(93-Day Mature Fodder)					
FDP.SA	53437	0.048	<0 01	54	0 002
FDP.SB	53437	"	<0 01	52	0 001
Sample ID	Study Number 54-91.2 Code No.	Incurred ¹⁴ C Level (ppm) *	(HPLC) ppm Found	% ¹⁴ C Extracted	ppm ¹⁴ C Found in Final Volume
(Sprayed Triazine- ¹⁴ C-CGA-152005)					
(0-Day Forage)					
FLT.SA	53405	3.30	1.69	100	1 30
(30-Day Forage)					
FFT SA	53406	0 029	<0.01	79	0 001
FFT.SB	53406	"	<0.01	86	<0.001
(46-Day Silage Stage Forage)					
FST.SA	53407	0.048	<0.01	101	0 001
FST SB	53407	"	<0 01	90	0.001
(93-Day Mature Fodder)					
FDT.SA	53408	0 009	<0.01	30	<0.001
FDT.SB	53408	"	<0.01	30	<0.001

* ¹⁴C incurred levels determined by combustion/LSC by Metabolism Department. Reference Lab Notebooks 4002 and 4045

** Sample results not available due to documented problems during workup or analysis.

COMMENTS Results are corrected for procedural recoveries <100%

TABLE IV. SUMMARY OF RESULTS FOR ¹⁴C-CGA-152005 TREATED CORN (Continued)

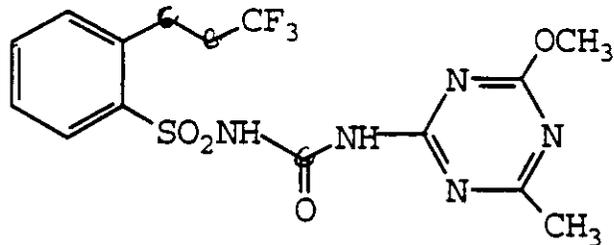
Sample ID	Study Number M91-168-007P Code No.	Incurred ¹⁴ C Level (ppm)*	(HPLC) ppm Found	% ¹⁴ C Extracted	ppm ¹⁴ C Found in Final Volume
(Injected Phenyl- ¹⁴ C-CGA-152005)					
(Mature Foliage)					
XFP IA	P91400161	0 308	0 032	99	0 032
XFP IB	P91400161	0 308	0.028	104	0 030
XFP IC	P91400161	0.308	0.033	96	0.031
(CV 9%)					
(Mature Stalk)					
SP IA	P91400078	0 195	<0 01	103	0 008
SP IB	P91400078	0 195	-NA-**	108	0 007
SP IC	P91400078	0 195	<0.01	108	0 006
(CV:14%)					
Sample ID	Study Number M91-168-008P Code No.	Incurred ¹⁴ C Level (ppm)*	(HPLC) ppm Found	% ¹⁴ C Extracted	ppm ¹⁴ C Found in Final Volume
(Injected Triazine- ¹⁴ C-CGA-152005)					
(Mature Foliage)					
XFT IA	P91400175	1.28	0 14	87	0 15
XFT IB	P91400175	1.28	0.14	90	0 15
XFT IC	P91400175	1.28	0.21	94	0 19
(CV:25%)					
(Mature Stalk)					
ST IA	P91400061	0.262	-NA-**	103	-NA-**
ST IB	P91400061	0 262	<0 01	134	0.006
ST IC	P91400061	0.262	<0 01	99	0.006
(Mature Grain)					
GT IA	P91400063	0 038	<0 01	70	<0 001
GT IB	P91400063	0 038	<0.01	70	<0.001
GT IC	P91400063	0.038	<0.01	68	<0.001

* ¹⁴C incurred levels determined by combustion/LSC by Metabolism Department. Reference Lab Notebooks 3955 and 3921

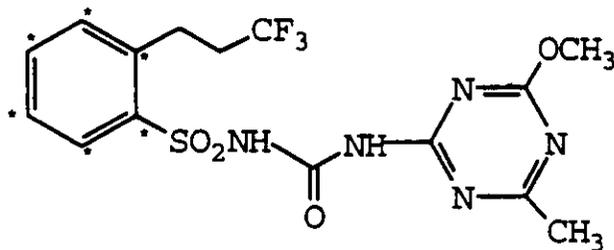
** Sample results not available due to documented problems during workup or analysis.

COMMENTS. Results are corrected for procedural recoveries <100%.

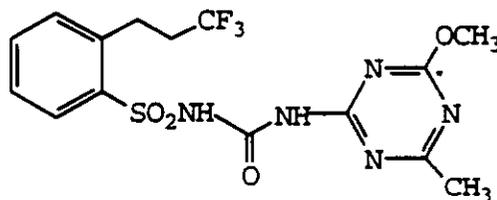
FIGURE 1. CHEMICAL NAMES AND STRUCTURES



CGA-152005
N-[[4-methoxy-6-methyl-1,3,5-triazin-2-yl]amino]carbonyl]-
2-(3,3,3-trifluoropropyl)-Benzenesulfonamide
CAS No 94125-34-5



Phenyl Label CGA-152005
N-[[4-methoxy-6-methyl-1,3,5-triazin-2-yl]amino]carbonyl]-
2-(3,3,3-trifluoropropyl)-[U-¹⁴C]-Benzenesulfonamide



Triazine Label CGA-152005
N-[[4-methoxy-6-methyl-1,3,5-[2-¹⁴C]-triazin-
2-yl]amino]carbonyl]-2-(3,3,3-trifluoropropyl)-
Benzenesulfonamide

FIGURE 2. SCHEMATIC DIAGRAM OF THE HPLC COLUMN SWITCHING SYSTEM

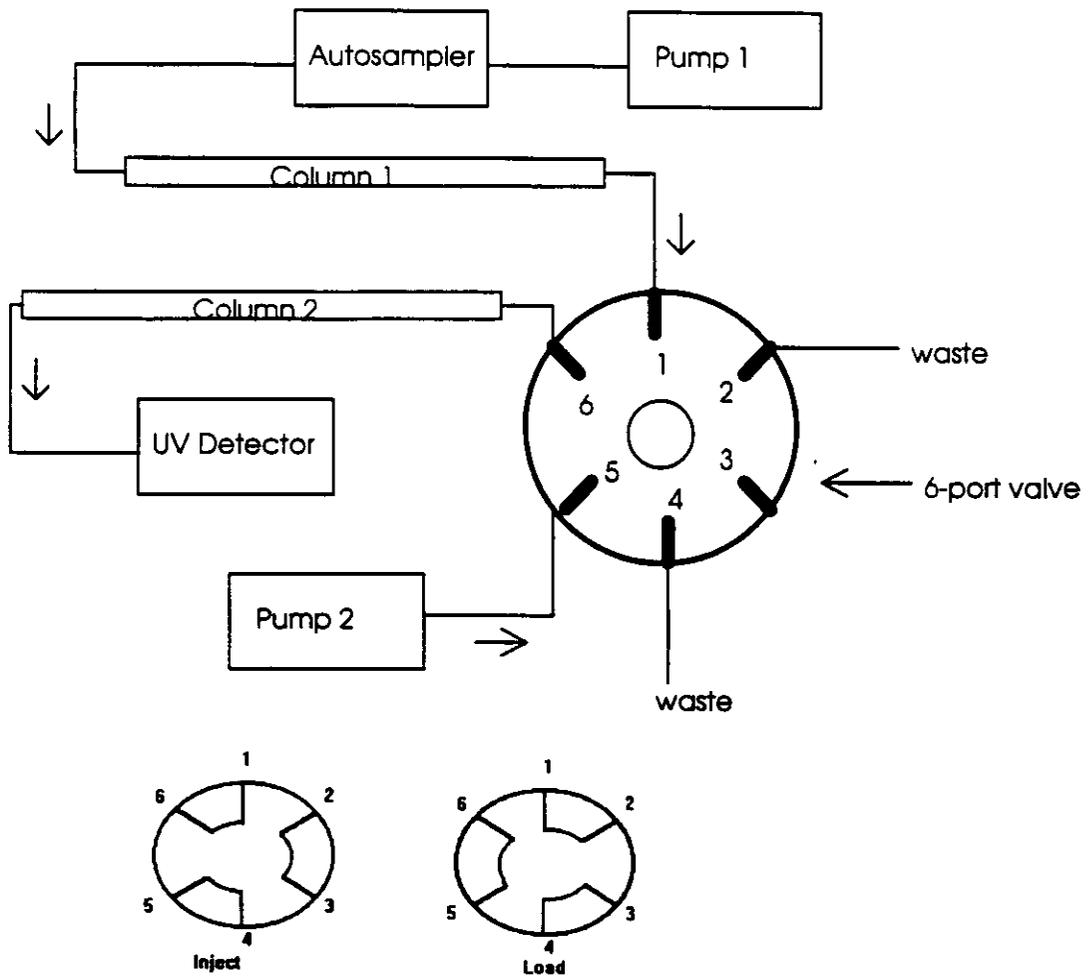
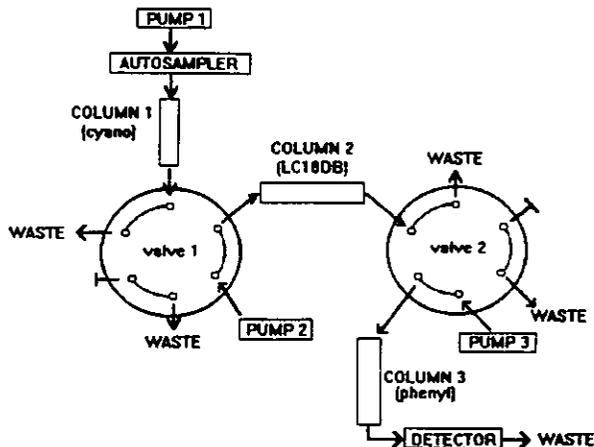
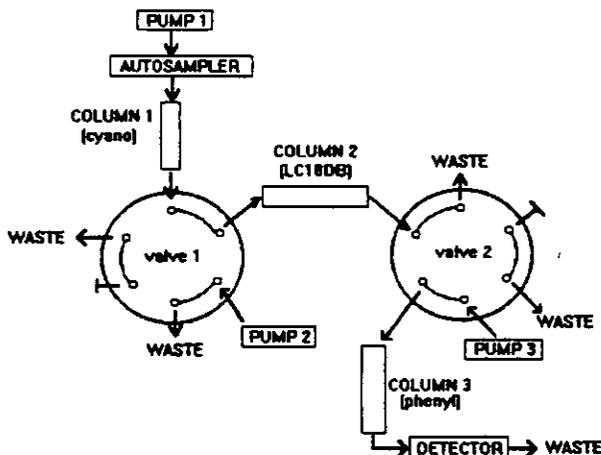


FIGURE 2a. SCHEMATIC DIAGRAM OF THE (CONFIRMATORY) HPLC TRIPLE COLUMN SWITCHING SYSTEM

POSITION A - INJECTION OF SAMPLE ON PREPARATORY COLUMN 1 (CYANO):



POSITION B - SWITCH FROM COLUMN 1 (CYANO) TO COLUMN 2 (LC18DB):



POSITION C - SWITCH FROM COLUMN 2 (LC18DB) TO COLUMN 3 (PHENYL):

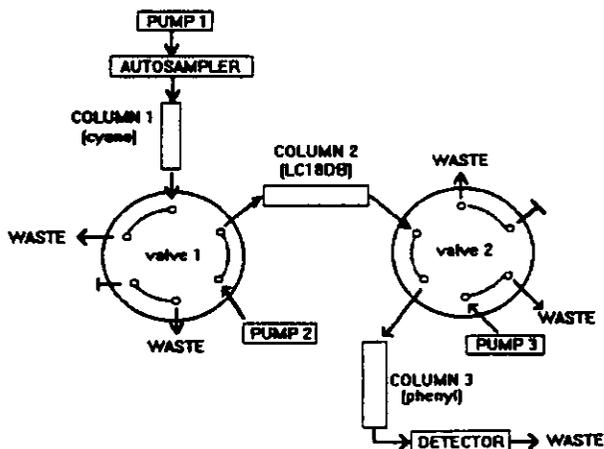


FIGURE 3. FLOW DIAGRAM FOR ANALYTICAL METHOD AG-590C:
SOLID SUBSTRATES

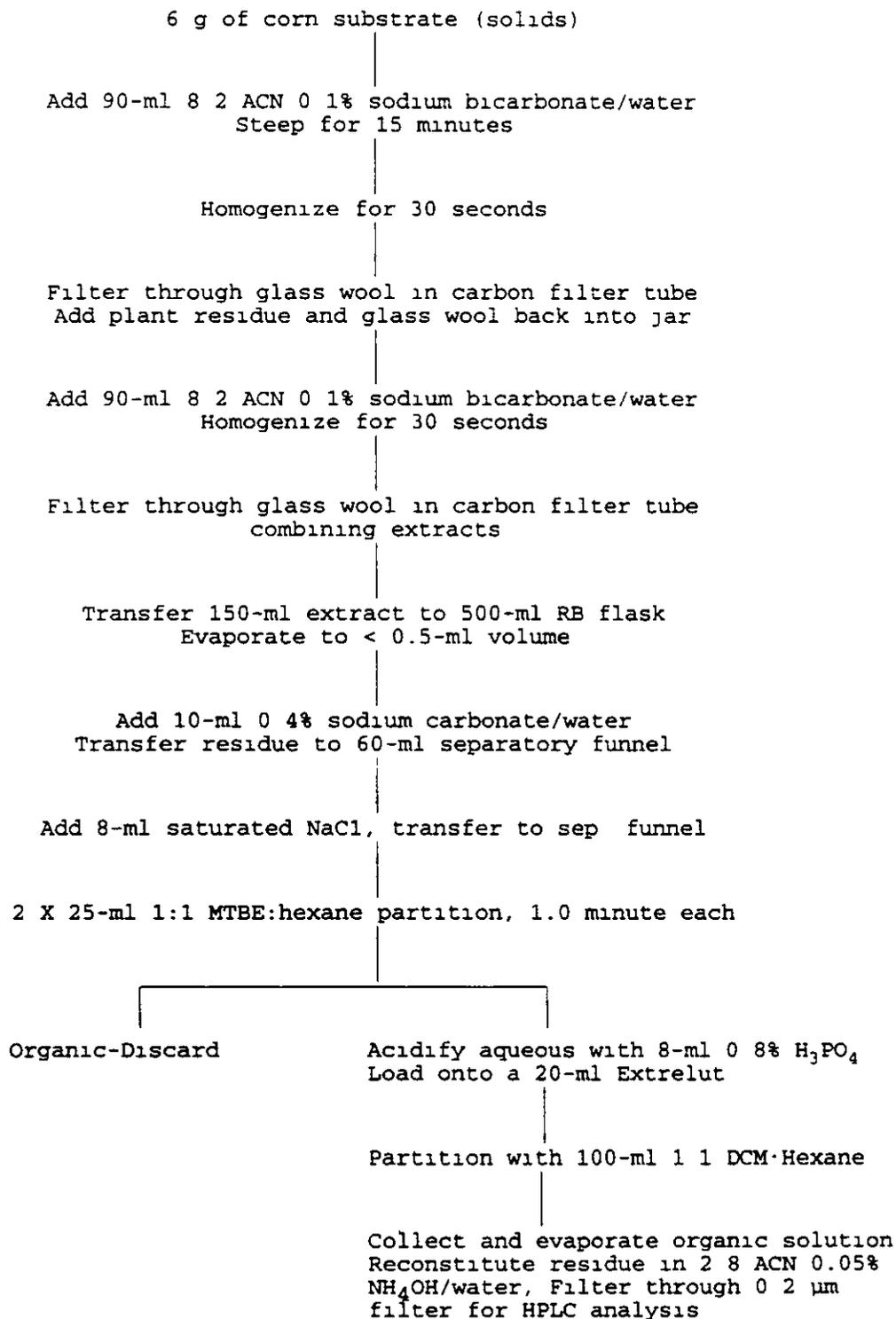


FIGURE 4. FLOW DIAGRAM FOR ANALYTICAL METHOD AG-590C:
OIL SUBSTRATES

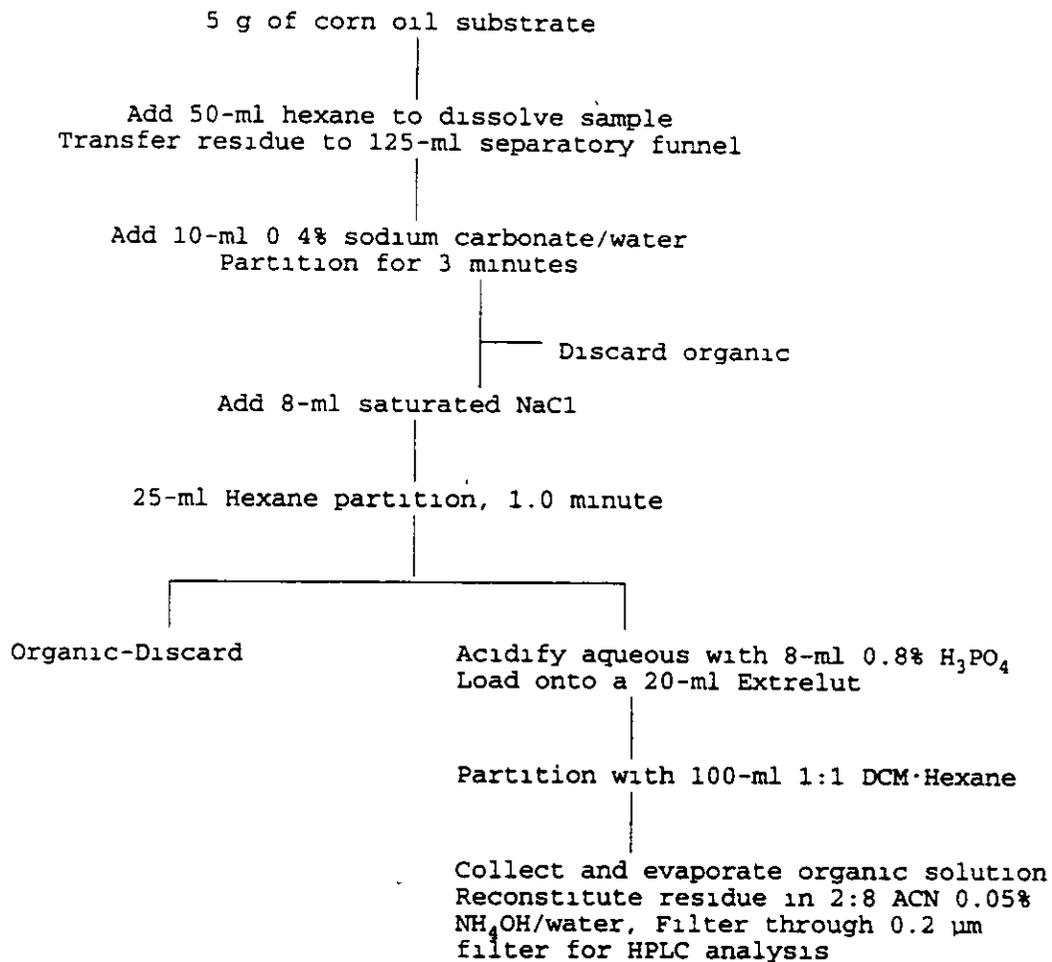
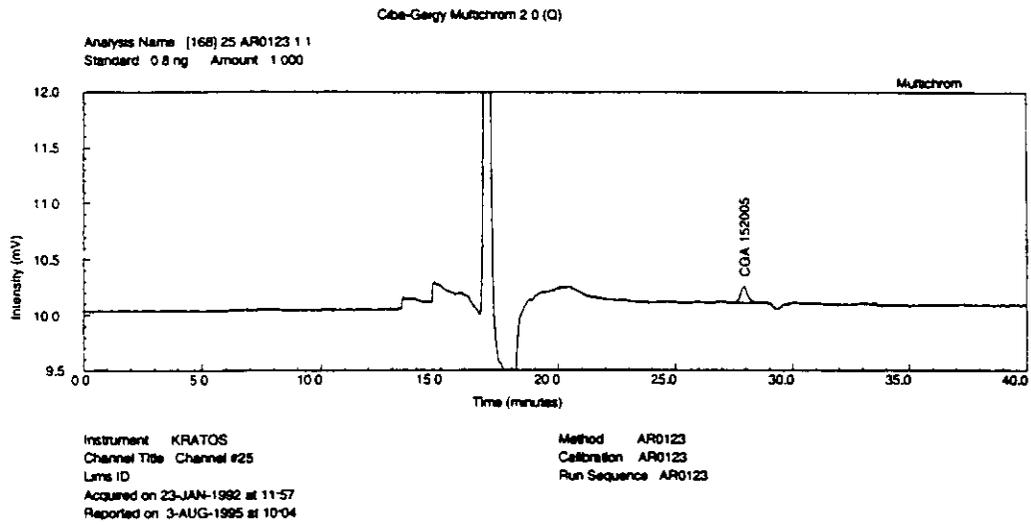
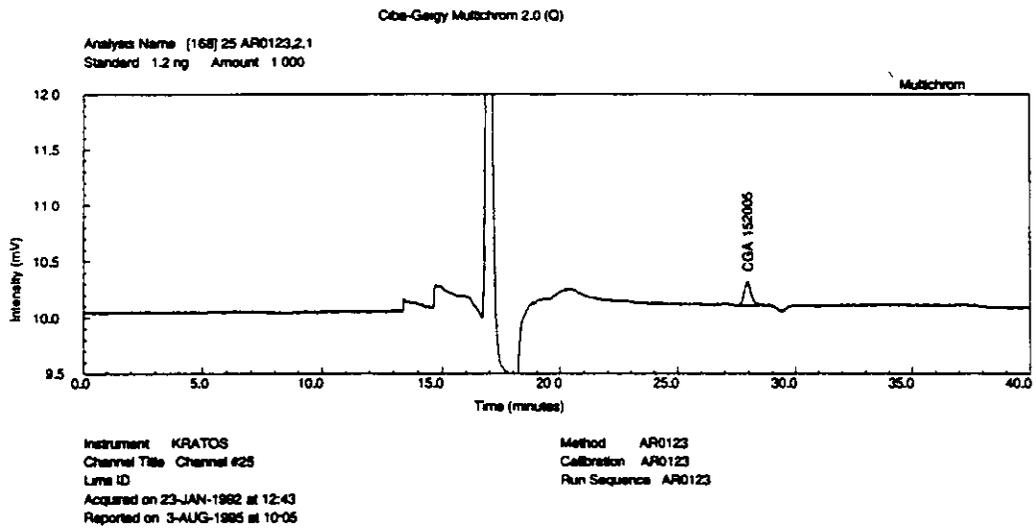


FIGURE 5. REPRESENTATIVE CHROMATOGRAMS FOR CGA-152005 STANDARDS

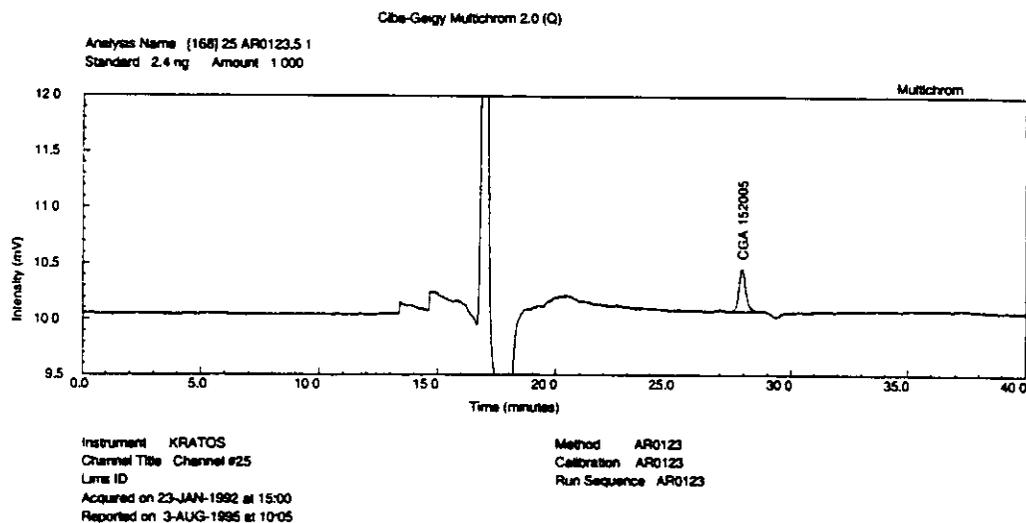


0.8 ng, CGA-152005 Standard

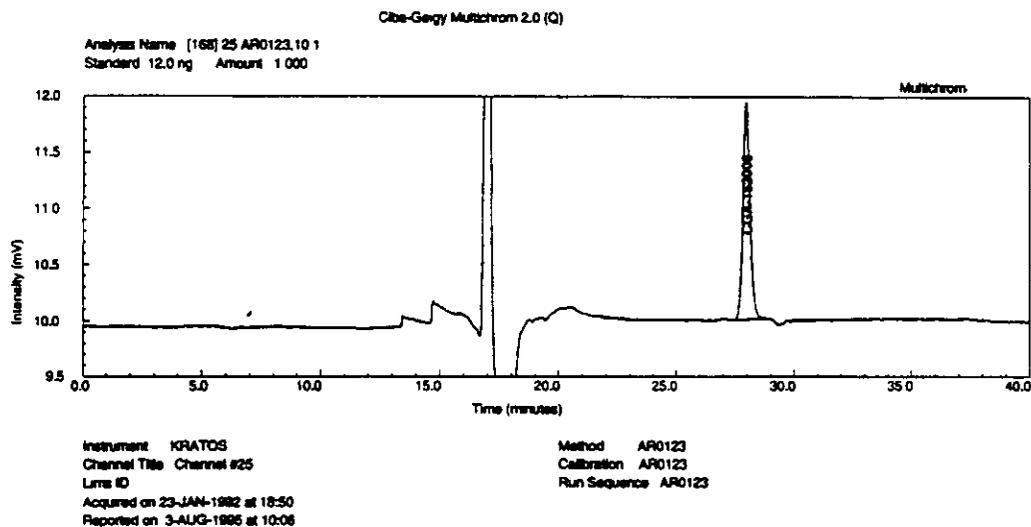


1.2 ng, CGA-152005 Standard

FIGURE 5. REPRESENTATIVE CHROMATOGRAMS FOR CGA-152005 STANDARDS (Continued)

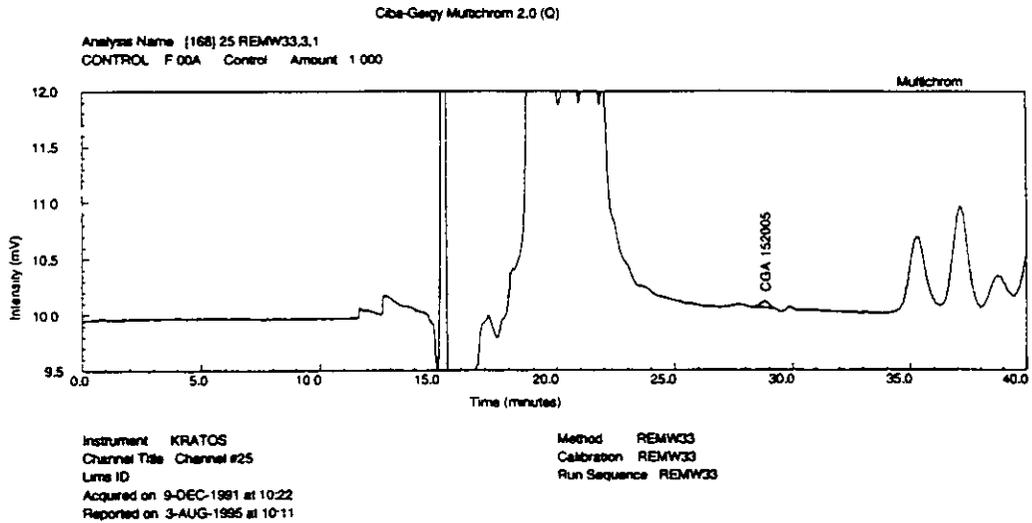


2.4 ng, CGA-152005 Standard

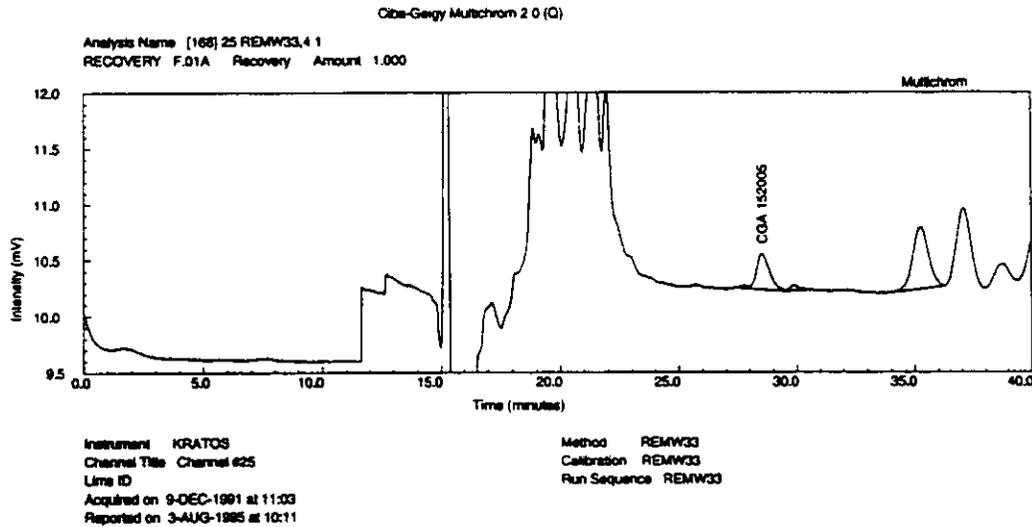


12 ng, CGA-152005 Standard

FIGURE 6 REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN FORAGE SAMPLES



(1)

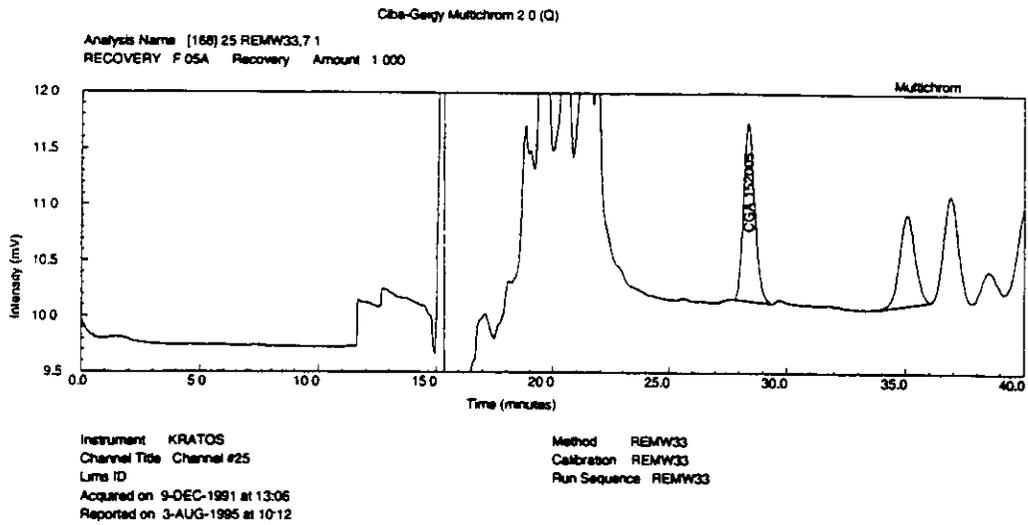


(2)

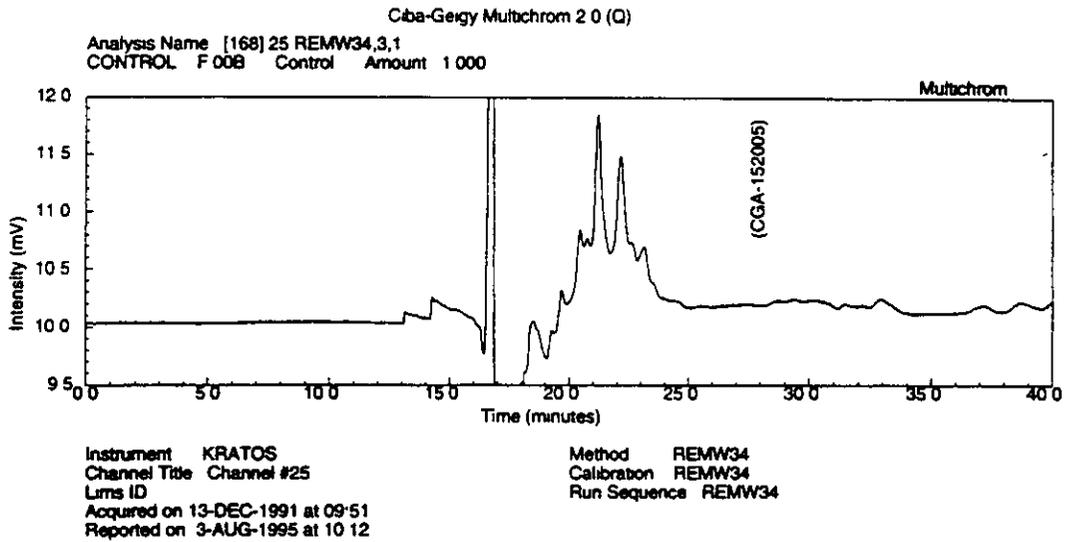
- 1 F 00A 0-day Corn Forage, 195 mg injected, 0.58 ng found, <0.01 ppm (0.003 ppm)
- 2 F 01A. 0-day Corn Forage + 0.01 ppm CGA-152005, 195 mg injected, 2.1 ng found; 0.011 ppm; 80% recovery

(Recovery results corrected for control values)

FIGURE 6. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN FORAGE SAMPLES (Continued)



(3)

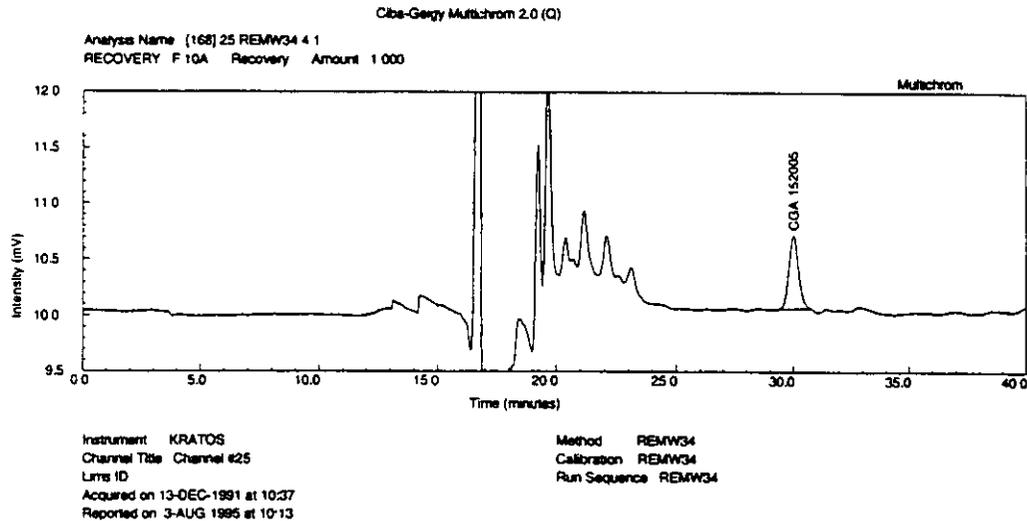


(4)

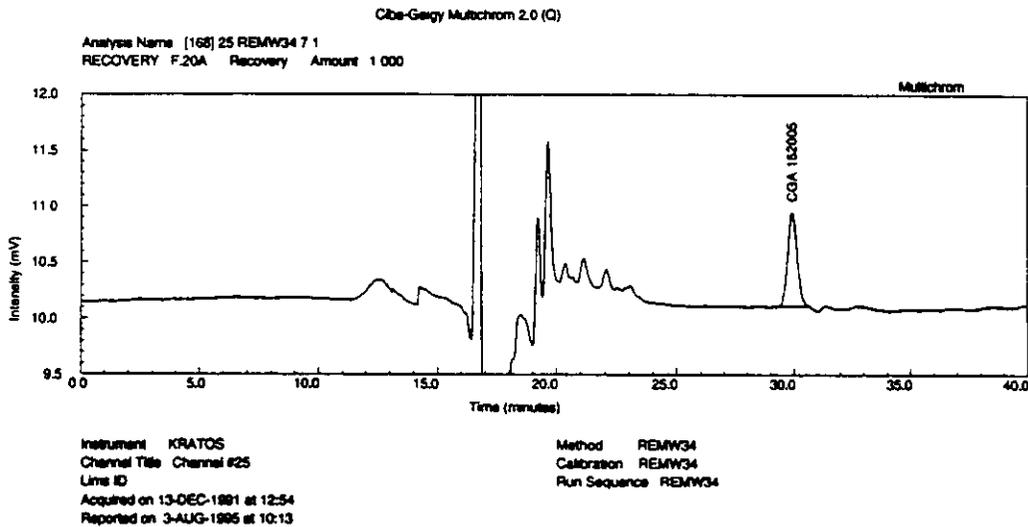
- 3. F 05A: 0-day Corn Forage + 0.05 ppm CGA-152005, 195 mg injected; 9.6 ng found; 0.049 ppm; 92% recovery
- 4. F 00B: 30-day Corn Forage, 195 mg injected, <0.8 ng found, <0.01 ppm

(Recovery results corrected for control values)

FIGURE 6. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN FORAGE SAMPLES (Continued)



(5)

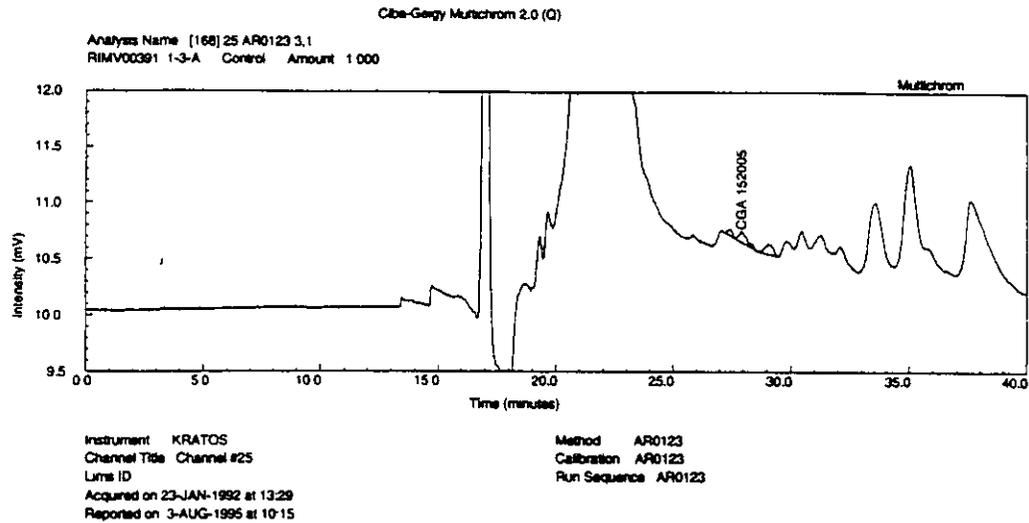


(6)

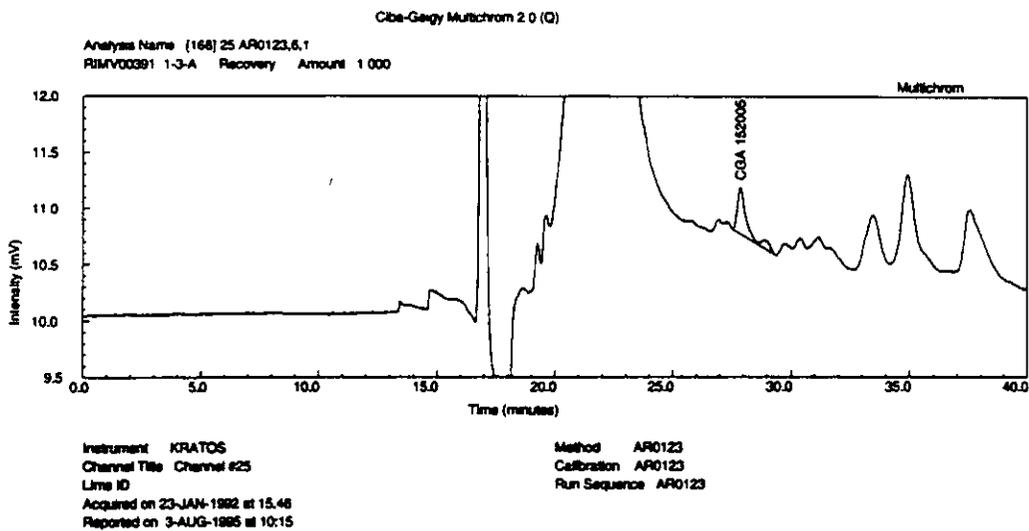
- | | | |
|---|-------|---|
| 5 | F 10A | 30-day Corn Forage + 0 10 ppm CGA-152005, 98 mg injected, 7.1 ng found; 0 073 ppm; 73% recovery |
| 6 | F 20A | 30-day Corn Forage + 0 20 ppm CGA-152005; 49 mg injected, 8 9 ng found; 0 18 ppm, 92% recovery |

(Recovery results corrected for control values)

FIGURE 7. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN FODDER SAMPLES



(1)

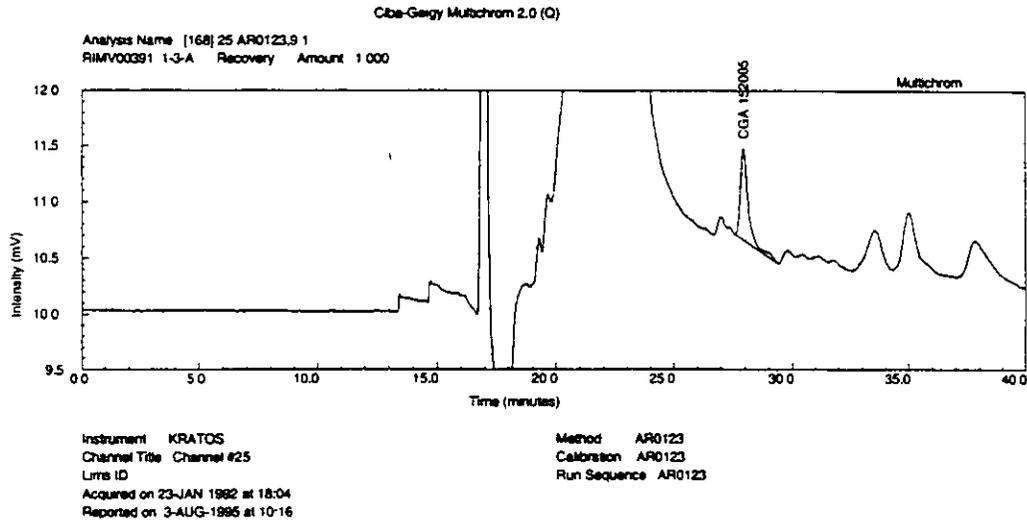


(2)

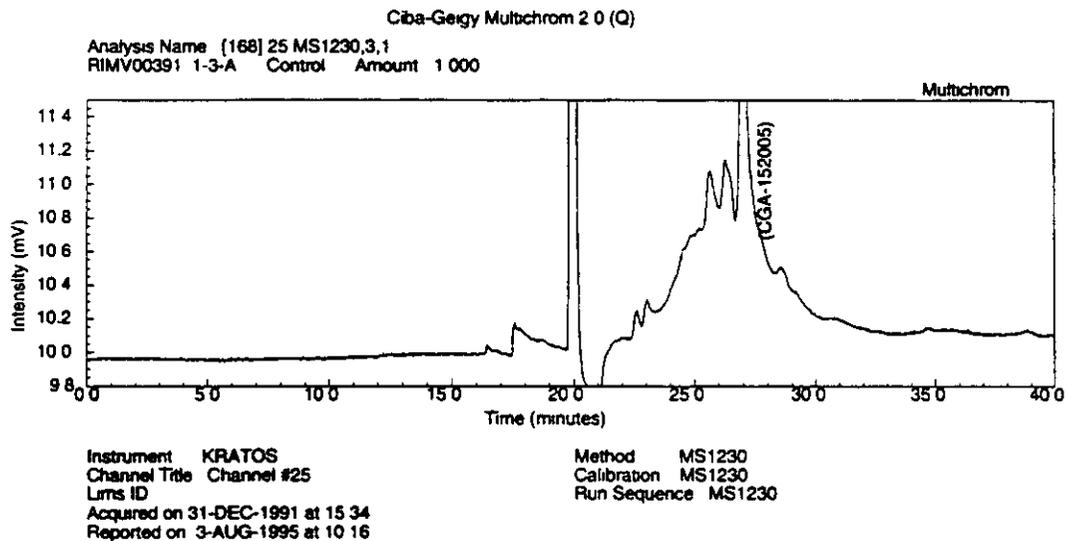
- 1 D 00A Corn Fodder, 198 mg injected; 0.48 ng found, <0.01 ppm (0.002 ppm)
- 2 D 01B. Corn Fodder + 0.01 ppm CGA-152005; 198 mg injected, 2.5 ng found, 0.013 ppm; 103% recovery

(Recovery results corrected for control values)

FIGURE 7. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN FODDER SAMPLES
(Continued)



(3)

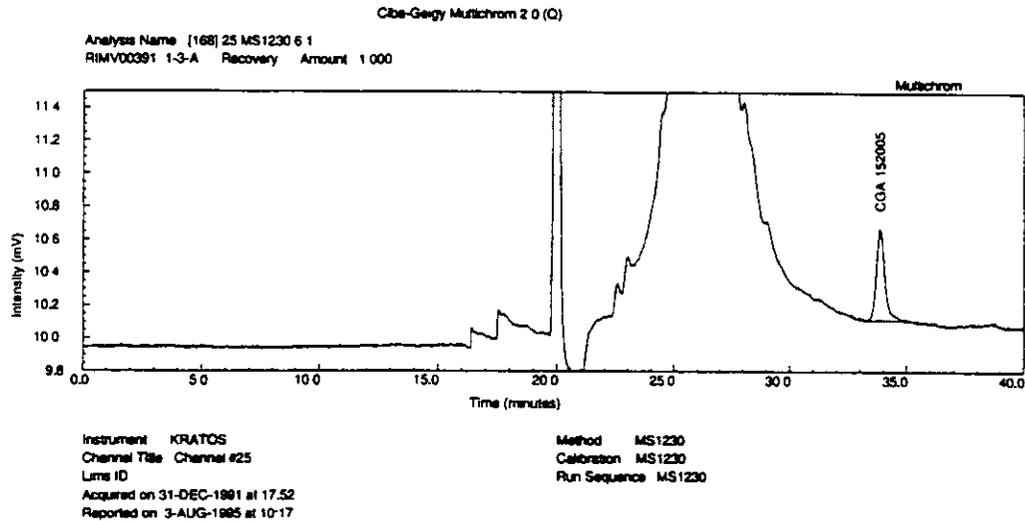


(4)

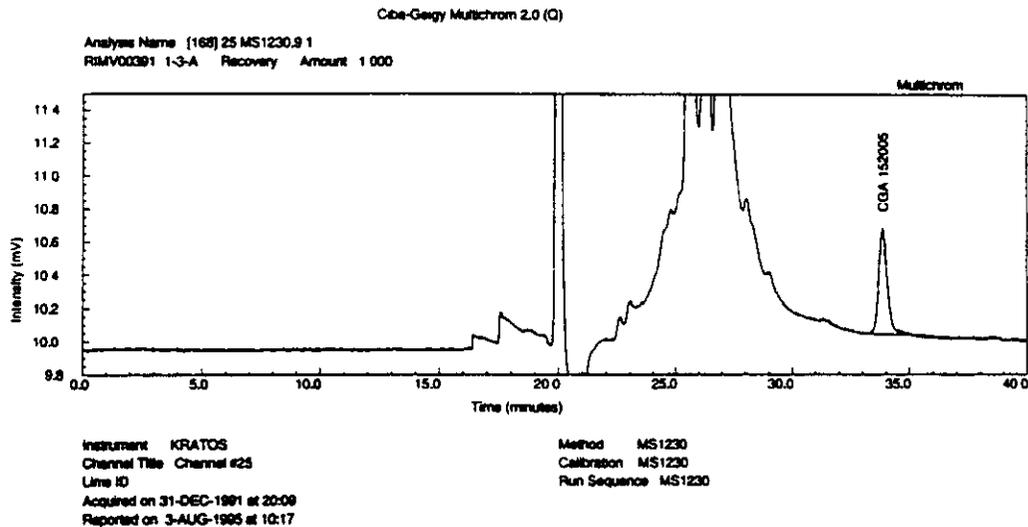
- 3 D 05B: Corn Fodder + 0 05 ppm CGA-152005; 99 mg injected, 5 0 ng found,
0 050 ppm; 96% recovery
- 4 D.00B Corn Fodder; 187 mg injected; <0 8 ng found, <0 01 ppm

(Recovery results corrected for control values)

FIGURE 7. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN FODDER SAMPLES (Continued)



(5)

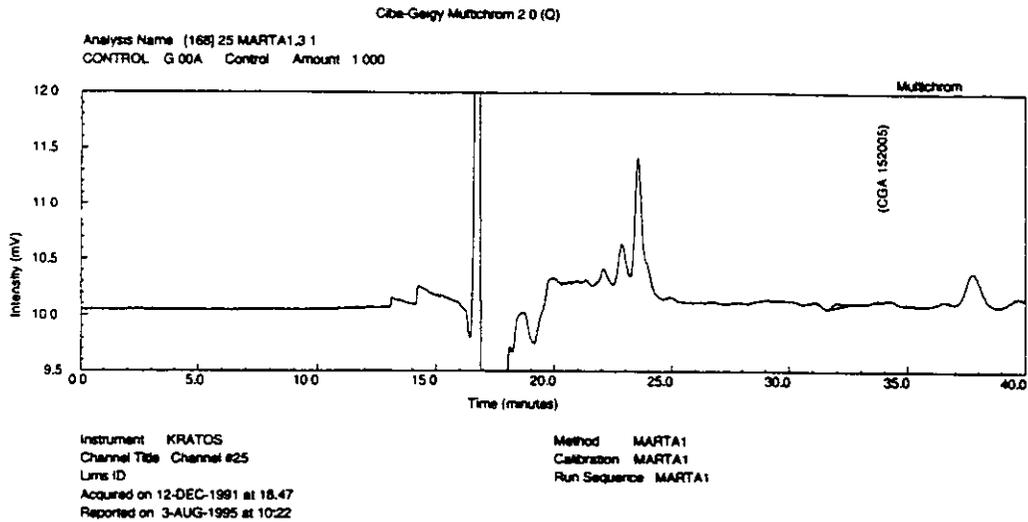


(6)

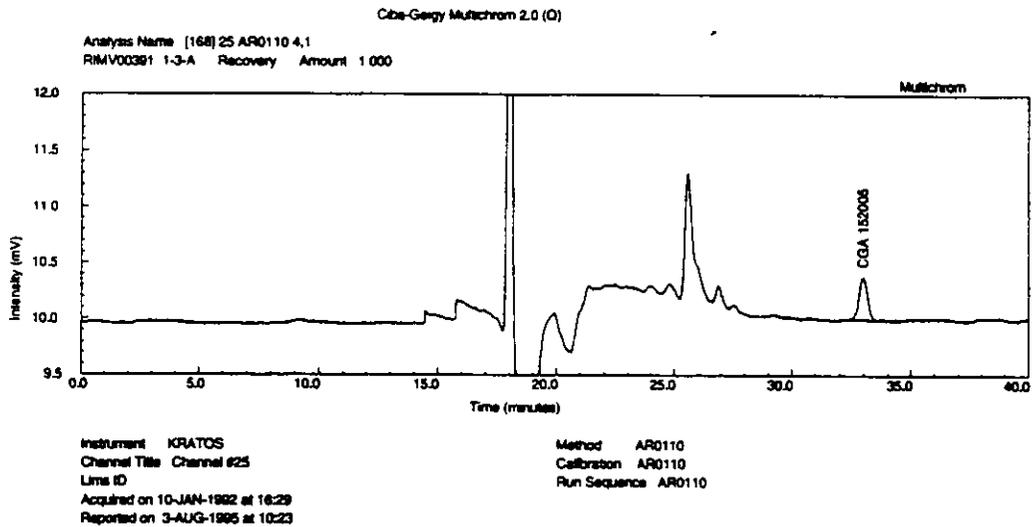
- 5 D.10B: Corn Fodder + 0.10 ppm CGA-152005, 90 mg injected, 8.9 ng found, 0.099 ppm, 99% recovery
- 6 D.20B: Corn Fodder + 0.20 ppm CGA-152005; 45 mg injected, 10 ng found, 0.22 ppm, 112% recovery

(Recovery results corrected for control values)

FIGURE 8. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN GRAIN SAMPLES



(1)

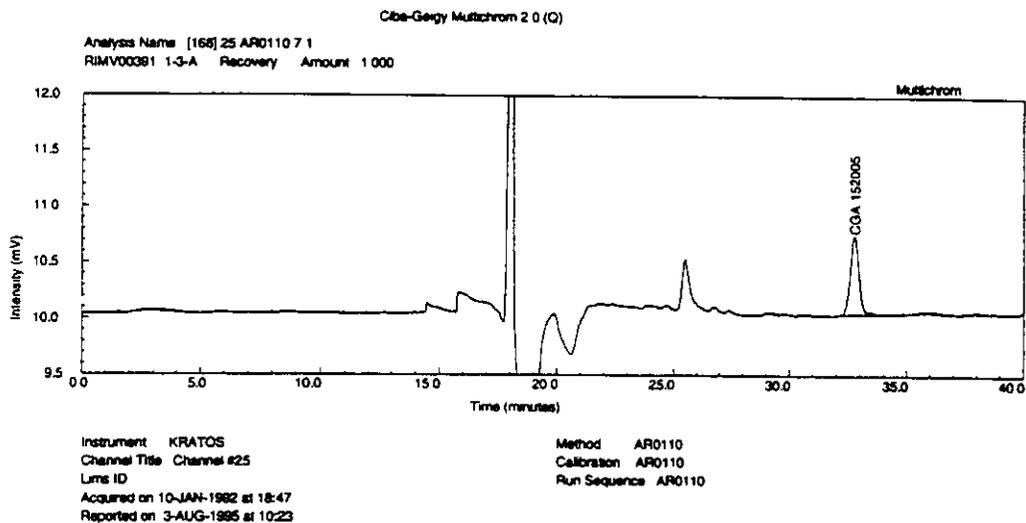


(2)

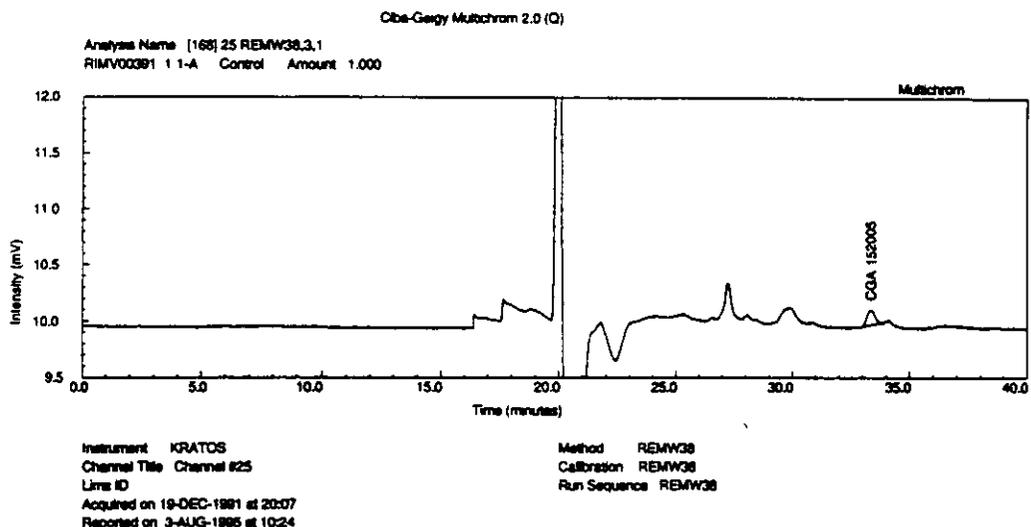
1. G 00A Corn Grain; 200 mg injected, <0.8 ng found; <0.01 ppm
2. G 01AR Corn Grain + 0.01 ppm CGA-152005, 200 mg injected, 2.4 ng found; 0.012 ppm, 120% recovery

(Recovery results corrected for control values)

FIGURE 8. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN GRAIN SAMPLES (Continued)



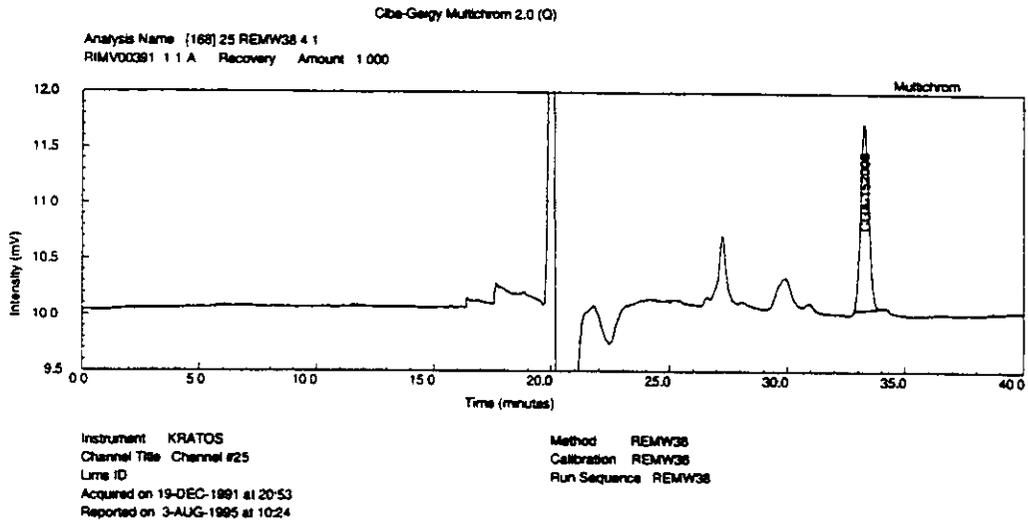
(3)



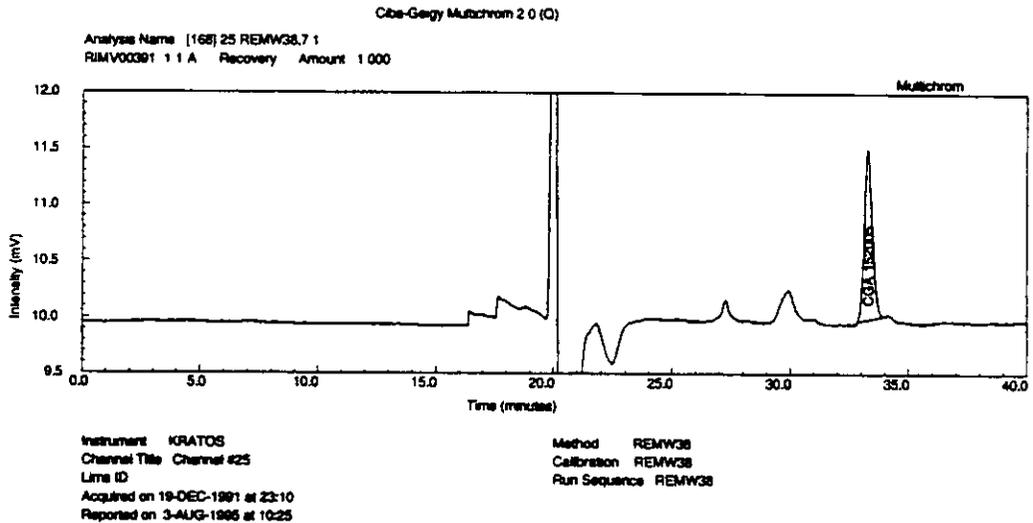
(4)

- 3 G 05AR Corn Grain + 0.05 ppm CGA-152005, 100 mg injected, 4.3 ng found, 0.042 ppm; 86% recovery
- 4 G 00BR: Corn Grain; 200 mg injected, 0.72 ng found, <0.01 ppm (0.004 ppm)
- (Recovery results corrected for control values)

FIGURE 8. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN GRAIN SAMPLES (Continued)



(5)

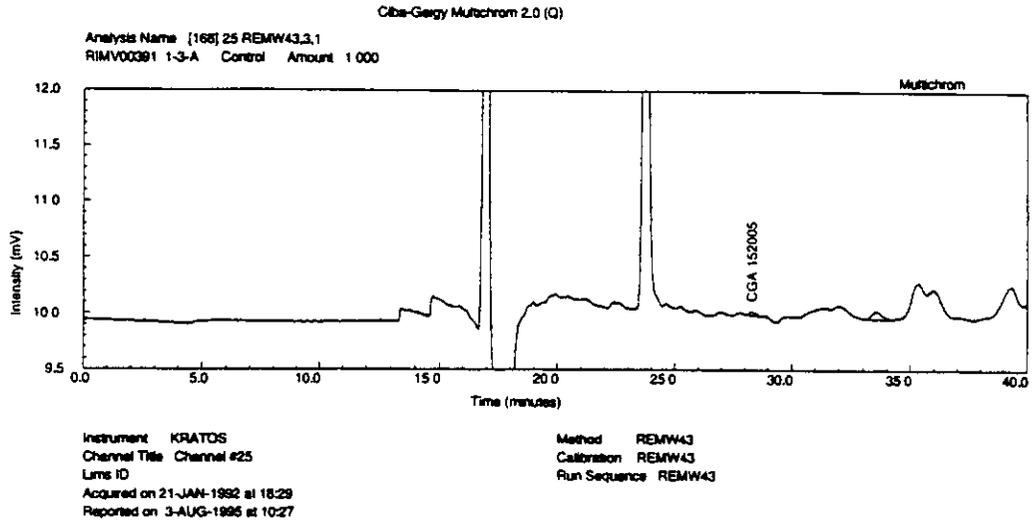


(6)

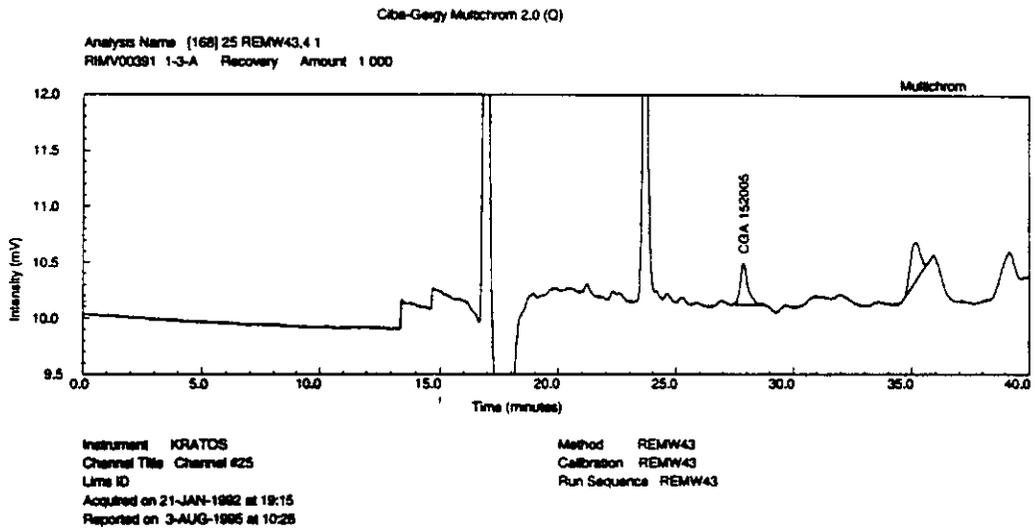
- 5 G 10AR Corn Grain + 0.10 ppm CGA-152005, 100 mg injected, 11 ng found,
0.11 ppm, 106% recovery
6. G 20AR Corn Grain + 0.20 ppm CGA-152005, 50 mg injected, 9.9 ng found,
0.20 ppm, 97% recovery

(Recovery results corrected for control values)

FIGURE 9. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN FLOUR SAMPLES



(1)

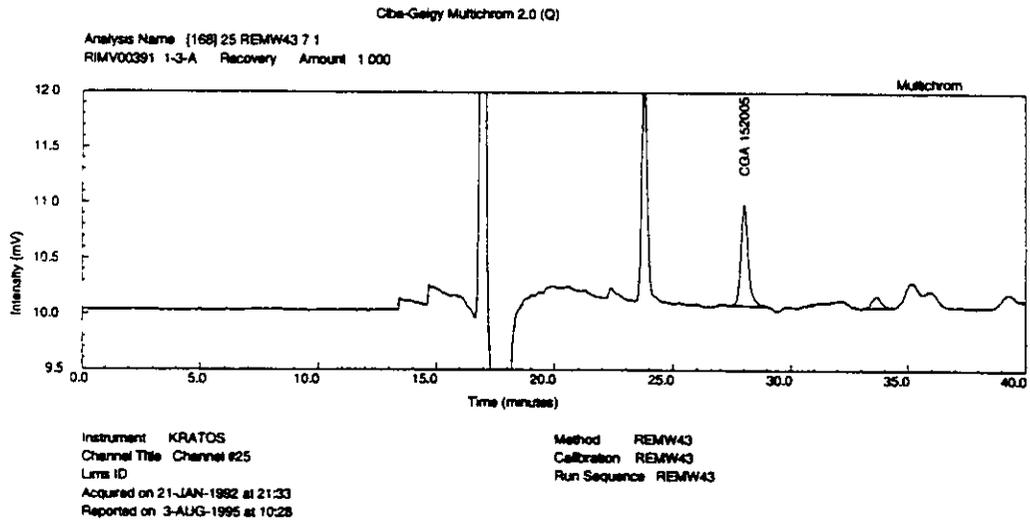


(2)

- 1 FLR 0 Corn Flour, 200 mg injected, <0.8 ng found, <0.01 ppm
- 2 FLR.01A: Corn Flour + 0.01 ppm CGA-152005, 200 mg injected; 2.1 ng found, 0.010 ppm, 97% recovery

(Recovery results corrected for control values)

FIGURE 9. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CONTROL CORN FLOUR SAMPLES (Continued).

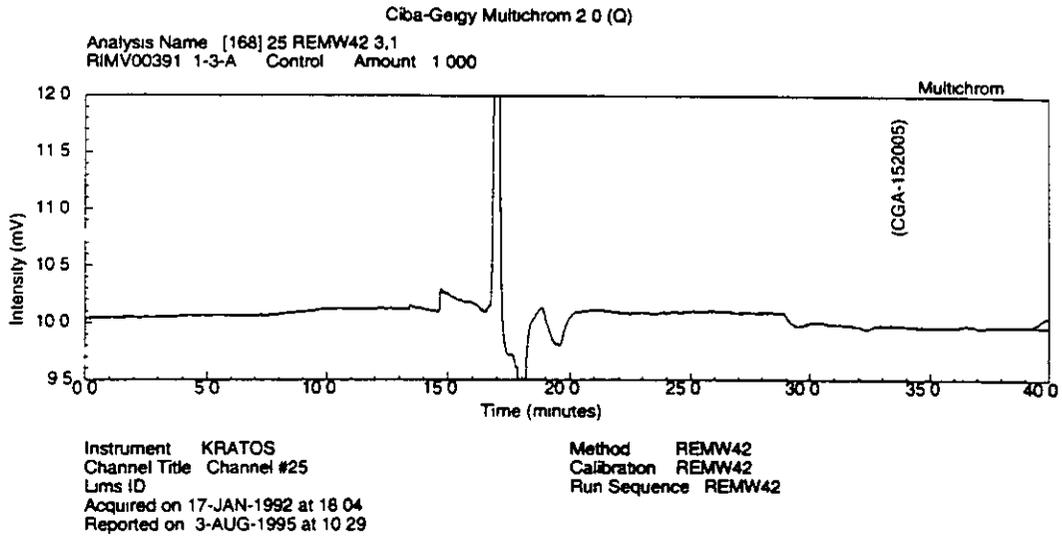


(3)

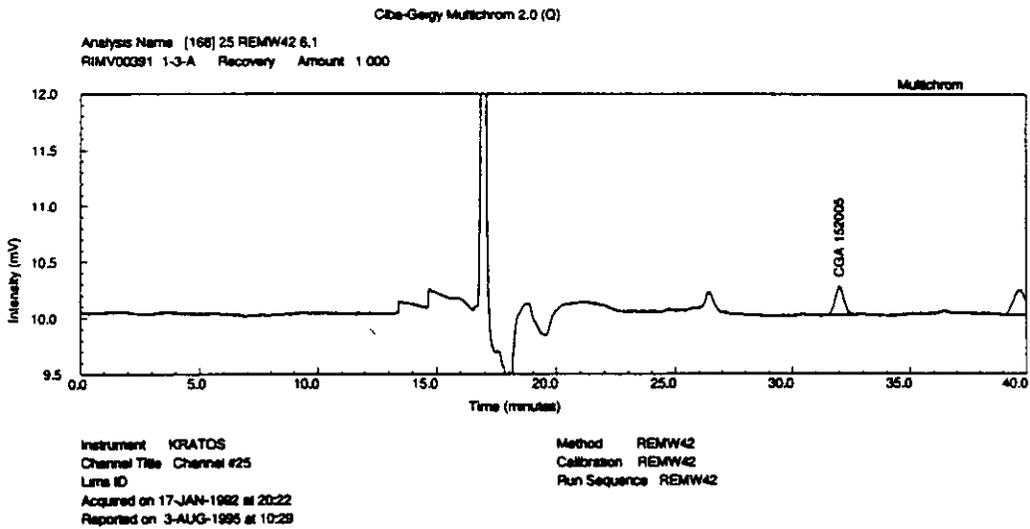
3 FLR 05 Corn Flour + 0.05 ppm CGA-152005; 100 mg injected; 5.2 ng found,
0.052 ppm; 102% recovery

(Recovery results corrected for control values)

FIGURE 10. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CORN OIL SAMPLES



(1)

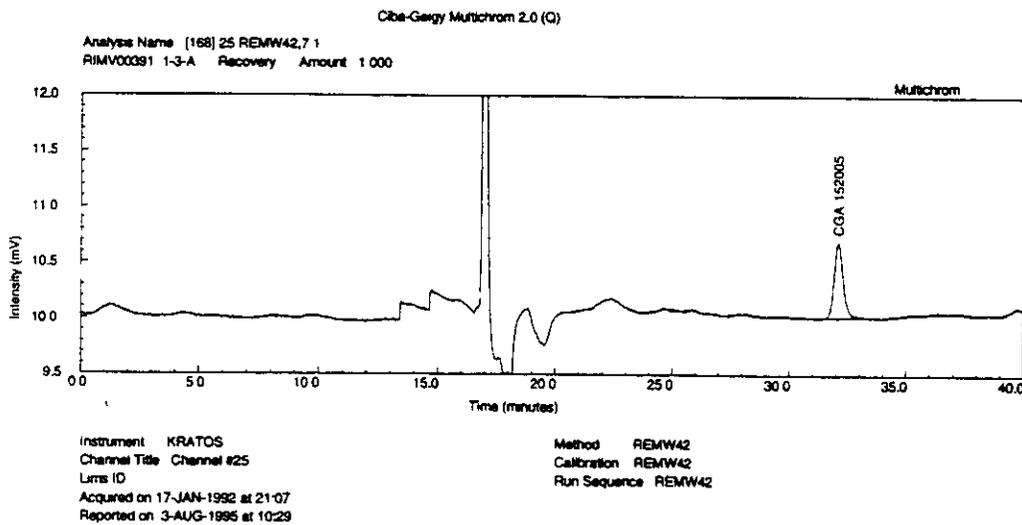


(2)

1. Oil.0 Corn Crude Oil, 200 mg injected, <0.8 ng found; <0.01 ppm
2. Oil 01B Corn Crude Oil + 0.01 ppm CGA-152005; 200 mg injected, 1.7 ng found, 0.009 ppm; 87% recovery

(Recovery results corrected for control values)

FIGURE 10. REPRESENTATIVE CHROMATOGRAMS FOR CONTROL AND FORTIFIED CORN OIL SAMPLES (Continued)

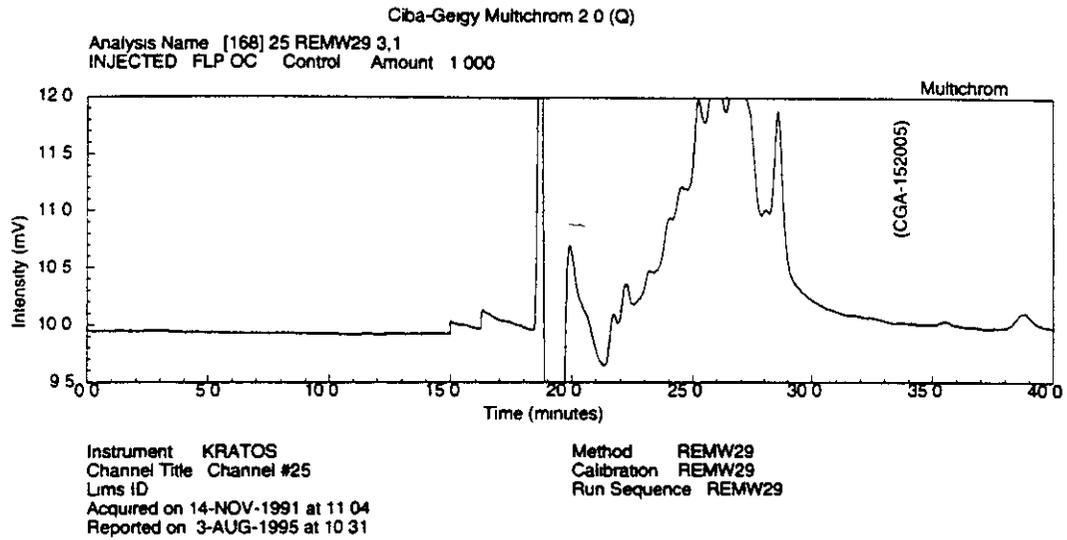


(3)

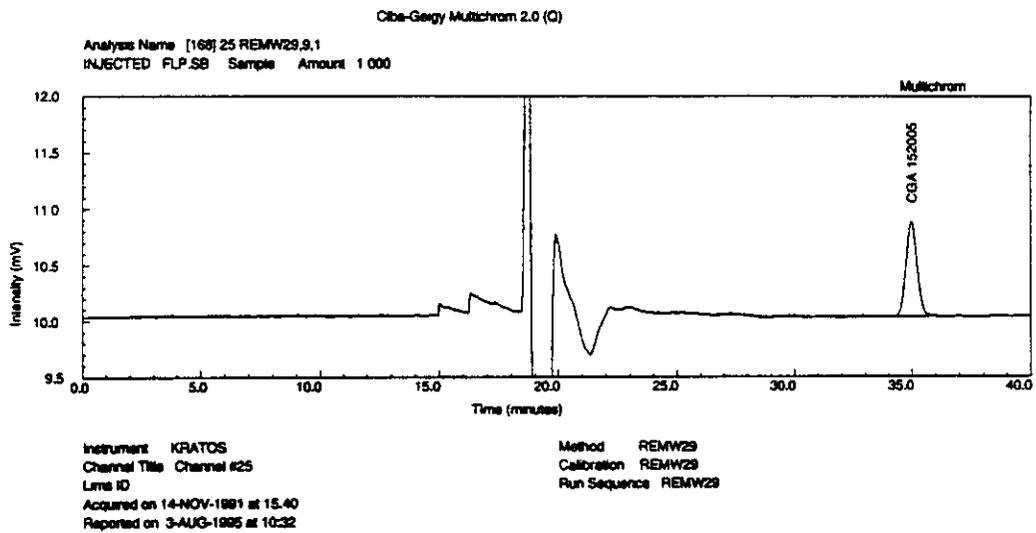
3 Oil 05A Corn Crude Oil + 0.05 ppm CGA-152005, 100 mg injected, 4.2 ng found, 0.042 ppm, 84% recovery

(Recovery results corrected for control values)

FIGURE 11. REPRESENTATIVE CHROMATOGRAMS FOR ¹⁴C-CGA-152005 TREATED CORN FORAGE SAMPLES



(1)

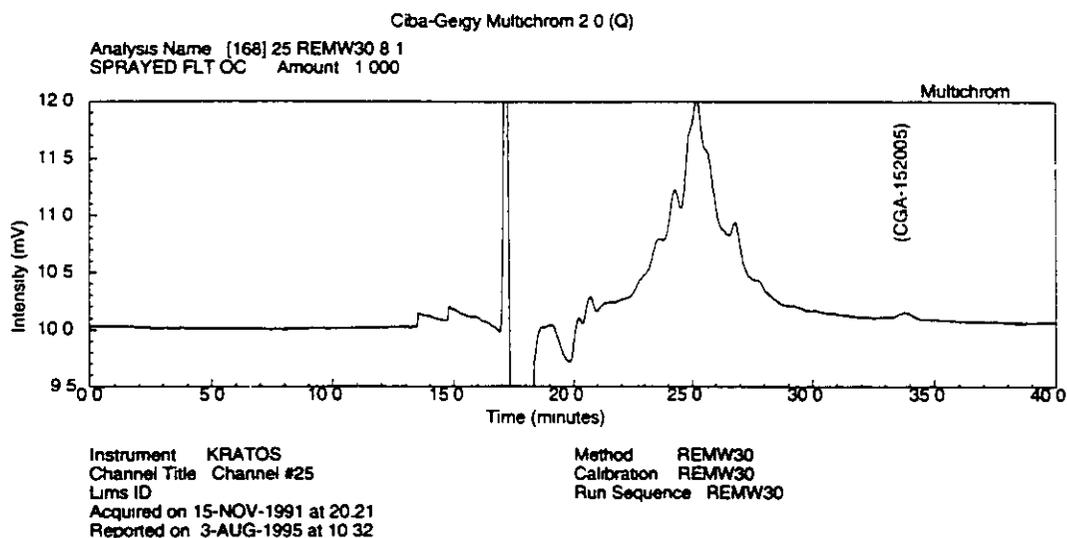


(2)

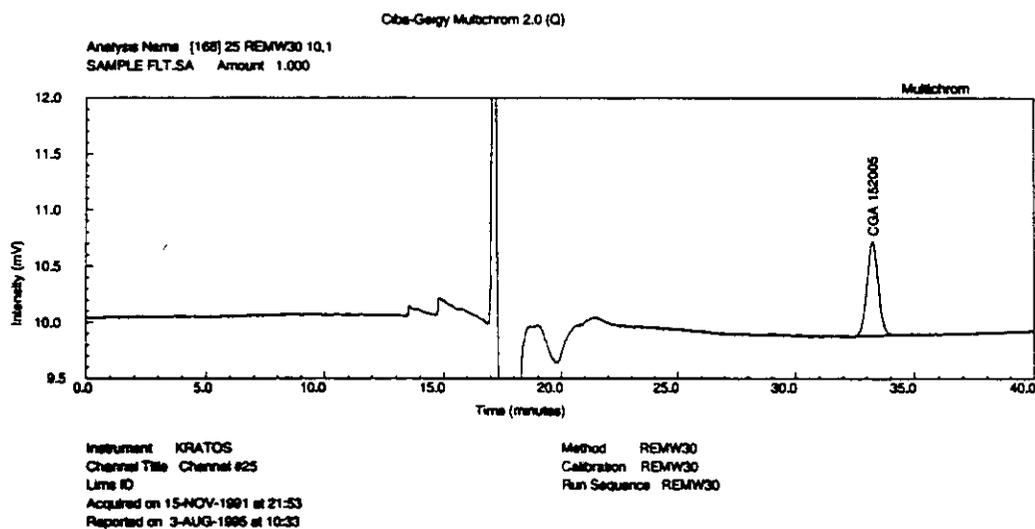
- 1 FLP OC 0-Day Corn Forage, 195 mg injected, <0.8 ng found; <0.01 ppm
- 2 FLP SB. 0-Day Corn Forage treated with phenyl-¹⁴C-CGA-152005, 3.9 mg injected; 6.0 ng found, 1.63 ppm

(Sample values corrected for procedural recoveries <100%)

FIGURE 11. REPRESENTATIVE CHROMATOGRAMS FOR ¹⁴C-CGA-152005 TREATED CORN FORAGE SAMPLES (Continued)



(3)

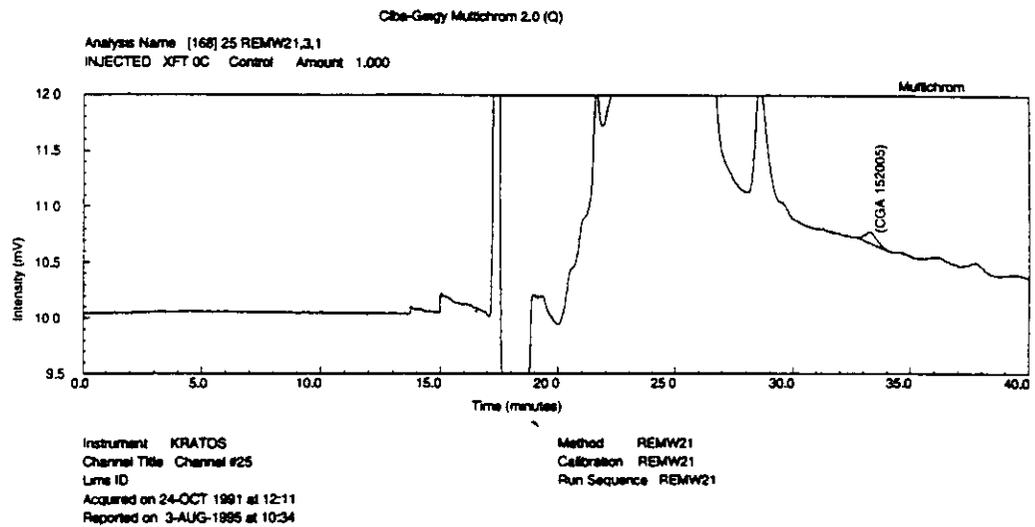


(4)

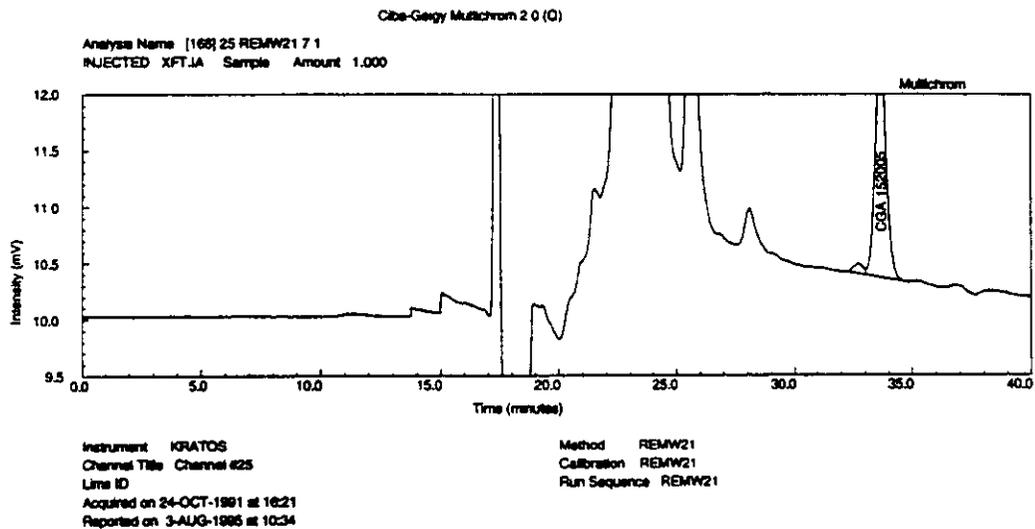
- 3 FLT.OC 0-Day Corn Forage; 195 mg injected, <0.8 ng found, <0.01 ppm
4 FLT.SA 0 Day Corn Forage treated with triazine-¹⁴C-CGA-152005, 3.9 mg injected, 6.2 ng found, 1.69 ppm

(Sample values corrected for procedural recoveries <100%)

FIGURE 12. REPRESENTATIVE CHROMATOGRAMS FOR ¹⁴C-CGA-152005 TREATED CORN FODDER SAMPLES



(1)

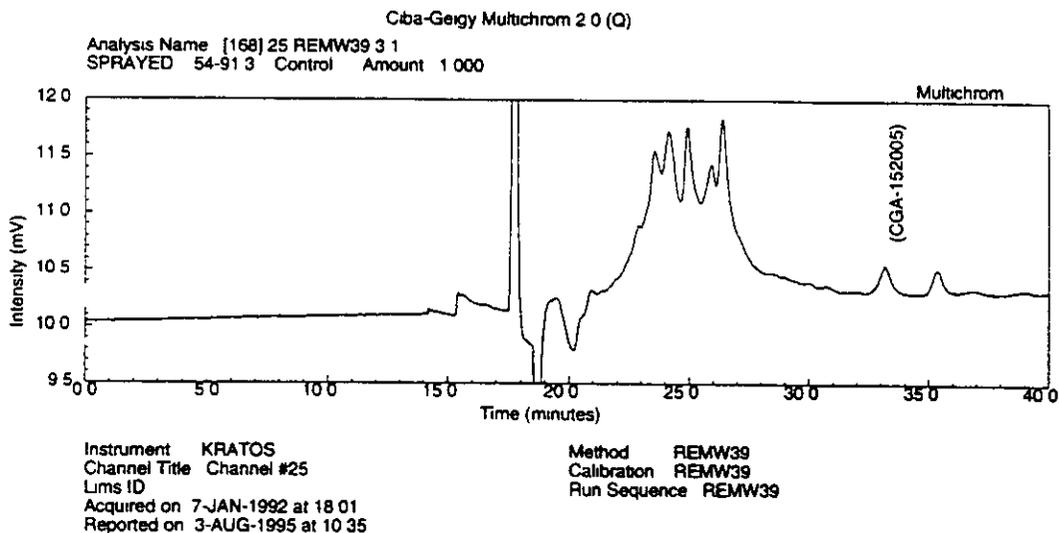


(2)

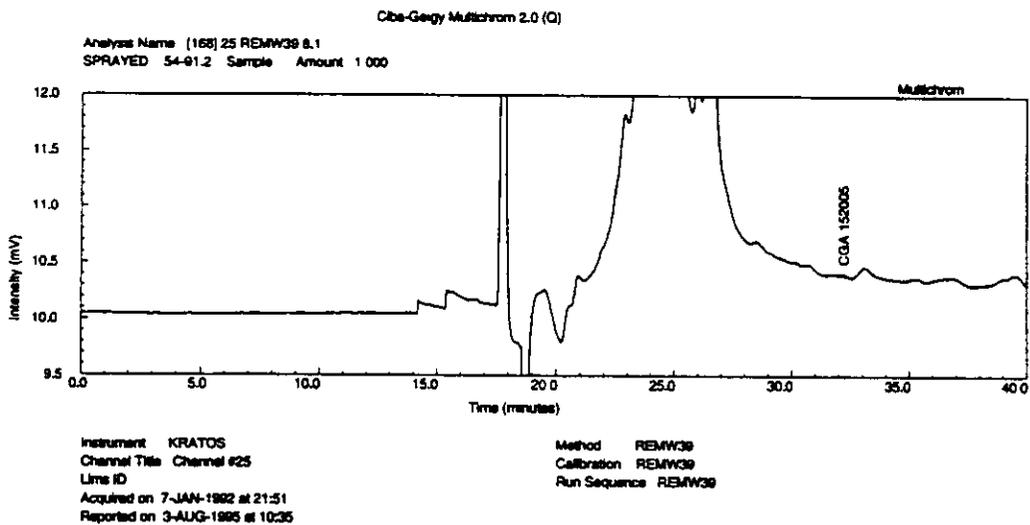
- 1 XFT OC Corn Fodder (Foliage) 186 mg injected, <0.8 ng found, <0.01 ppm
- 2 XFT.IA Corn Fodder (Foliage) treated with injected triazine-¹⁴C-CGA-152005, 96 mg injected, 11 ng found, 0.14 ppm

(Sample values corrected for procedural recoveries <100%)

FIGURE 12. REPRESENTATIVE CHROMATOGRAMS FOR ¹⁴C-CGA-152005 TREATED CORN FODDER SAMPLES (Continued)



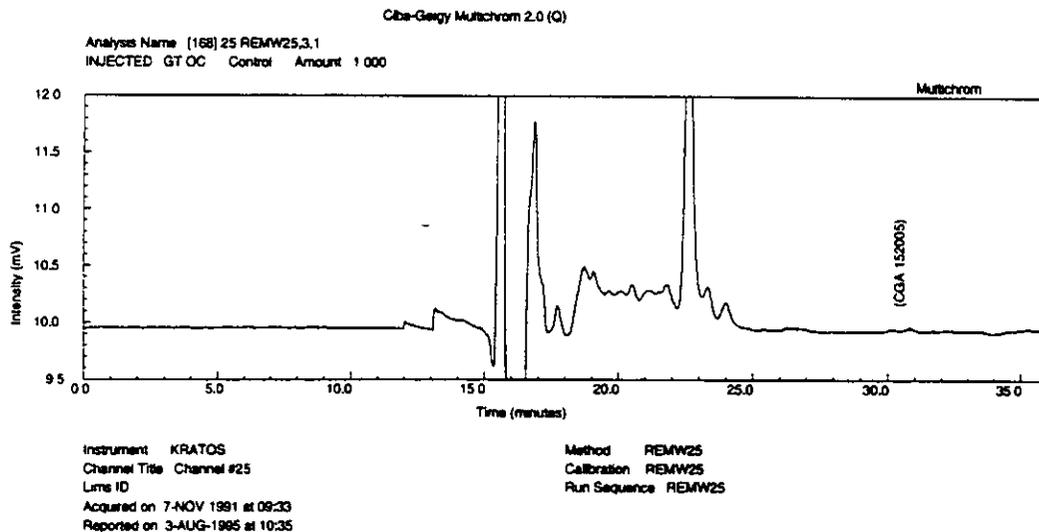
(3)



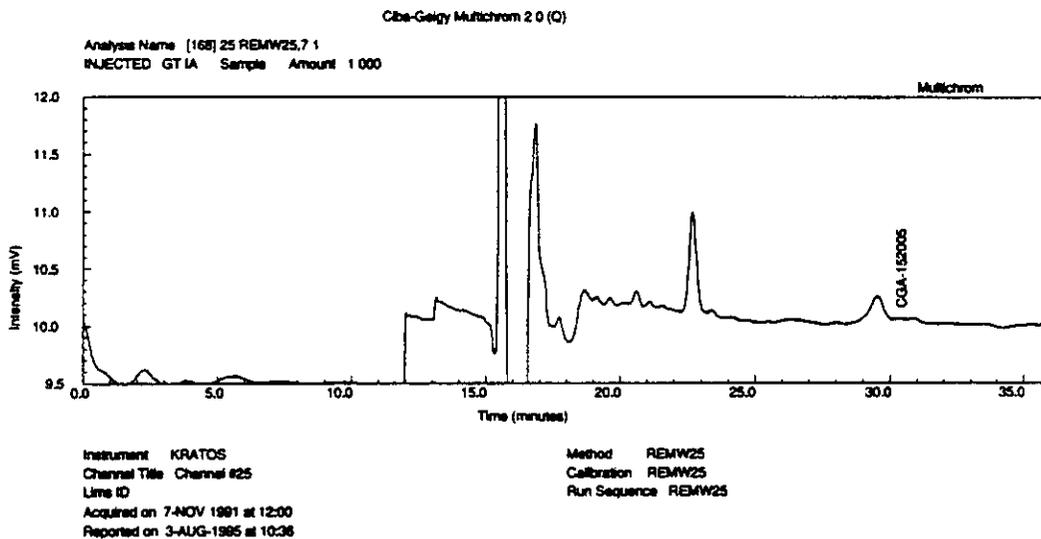
(4)

- 3 FDP OC: Corn Fodder 184 mg injected; <0.8 ng found, <0.01 ppm
4 FDP SA: Corn Fodder treated with phenyl-¹⁴C-CGA-152005, 197 mg injected, 0.40 ng found; <0.01 ppm

FIGURE 13. REPRESENTATIVE CHROMATOGRAMS FOR ¹⁴C-CGA-152005 TREATED CORN GRAIN SAMPLES



(1)

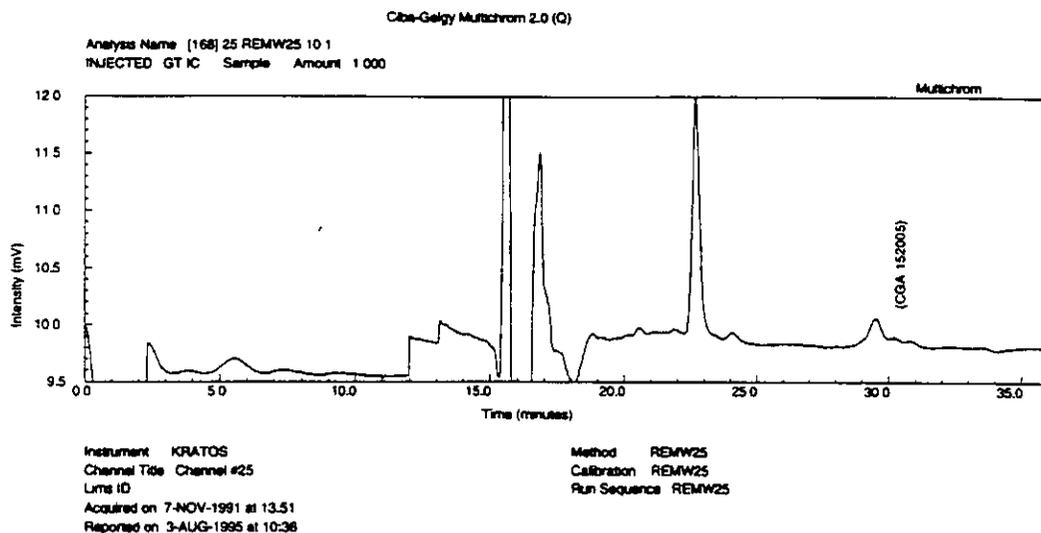


(2)

- | | | |
|---|-------|---|
| 1 | GT OC | Corn Grain, 200 mg injected; <0.8 ng found, <0.01 ppm |
| 2 | GT IA | Corn Grain; treated with injected triazine- ¹⁴ C-CGA-152005, 200 mg injected, <0.8 ng found; <0.01 ppm |

(Sample values corrected for procedural recoveries <100%)

FIGURE 13. REPRESENTATIVE CHROMATOGRAMS FOR ¹⁴C-CGA-152005 TREATED CORN GRAIN SAMPLES (Continued)



(3)

3 GT IC: Corn Grain, treated with injected triazine-¹⁴C-CGA-152005, 200 mg injected, <0 8 ng found, <0 01 ppm

(Sample values corrected for procedural recoveries <100%)

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1. T. L. Oakes, Analytical Method AG-590B, "Analytical Method for the Determination of CGA-152005 in Crops by High Performance Liquid Chromatography with Column Switching Including Validation Data."
2. D. Campbell and T. Oakes, Analytical Method AG-642, "Analytical Method for the Confirmation of Residues of CGA-152005 in Crops and Animal Substrates by Column Switching High Performance Liquid Chromatography "
3. R. E. M. Wurz, Analytical Method AG-590A, "Analytical Method for the Determination of CGA-152005 in Crops by High Performance Liquid Chromatography with Column Switching Including Validation Data." MRID No 43159349
4. Cindy Lochhaas, ABC Laboratories Report #42819, "Independent Laboratory Confirmation of Ciba Analytical Method No. AG-590B ('Analytical Method for the Determination of CGA-152005 in Crops by High Performance Liquid Chromatography with Column Switching Including Validation Data')."
5. Rolando Perez and Thomas Schreier, ADPEN Report #901-93-0108-002, "Independent Laboratory Confirmation of Ciba Analytical Method AG-590 ('Analytical Method for the Determination of CGA-152005 in Crops by High Performance Liquid Chromatography with Column Switching Including Validation Data') " MRID No. 431593515.
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7. R. Rezaaiyan, ABR-93048, "Uptake and Metabolism of CGA-152005 in Greenhouse Grown Corn After Spray Treatment with Phenyl-¹⁴C-CGA-152005 and Triazine-¹⁴C-CGA-152005." MRID No. 43159334
8. R. Rezaaiyan, ABR-93047, "Uptake and Metabolism of CGA-152005 in Field Grown Corn After Spray Treatment with Phenyl-¹⁴C-CGA-152005 and Triazine-¹⁴C-CGA-152005." MRID No. 43159336
9. R. E. M. Wurz, Residue Test Report RI-MV-003-91 No. 1. MRID No. 43159350