CYANAMID

American Cyanamid Company
Agricultural Research Division
Princeton, New Jersey 08543-0400

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Regulatory Affairs

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Protocol Number:	PR92PT01		
Project Number:	0471		
CL Numbers:	CL 92,553 CL 202,347	**Approved By:**	
Reported By:	S. Witkonton		
Work Done By:	ChemAlysis, Inc.	J. E. Boyd	921229 Date

TITLE:

CL 92,553 (pendimethalin): Validation of Method M 2243 for the Determination of CL 92,553 and CL 202,347 Residues in Canola Forage, Hay and Seed at ChemAlysis, Inc.

PURPOSE:

To validate Cyanamid Method M 2243 for the recovery of CL 92,553 and CL 202,347 residues in canola green forage, hay and seed.

SUMMARY:

ChemAlysis, Inc. validated the Cyanamid Method M 1930.01 (reissued as M 2243, See Appendix A) as written according to the protocol PR92PT01 (See Appendix B). A copy of the ChemAlysis report is attached as Appendix F.

Cyanamid method M 2243 is satisfactory for the determination of CL 92,553 and CL 202,347 residues in canola green forage, hay and seed with a validated sensitivity of 0.05 ppm for each compound and commodity. Recoveries were run by fortifying untreated samples with solutions of analytical grade standards (see Appendix F) and then processing them through the method. Summaries of CL 92,553 and CL 202,347 recoveries are presented in Tables 4A and 4B, respectively, of Appendix F. Detailed analytical data for recoveries of CL 92,553 and CL 202,347 can be found in Table 1A to 3B of Appendix F. Representative chromatograms from these analyses are shown in Appendix A (Method M 2243).

Recoveries were run in duplicate at fortifications of 0.05, 0.10, and 0.5 ppm for each compound. The recoveries of CL 92,553 and CL 202,347 averaged (± standard deviations) 94% ± 16% and 95% ± 19, respectively, for green forage; 87% ± 11% and 80% ± 8%, respectively, for hay; and 94% ± 4% and 94% ± 9%, respectively, for seed.

STABILITY OF TEST SUBSTANCES

CL 92,553 has been test and proved to be stable for 5 years (APRB Report #91) and CL 202,347 for 11 years (certification #91-ASA-040). The solutions of both compounds in organic solvents (iso-octane and ethyl acetate) have been proven to be stable for up to 6 months (AC8452:11).

SOLUBILITY OF TEST SUBSTANCES

CL 92,553 has been reported by C. Martin to be very soluble (up to 0.897 gm/mL) in ethyl acetate (Cyanamid Report F-816, 1985-1987, Title: PROWL Technical Solubility in Various Solvents and Solvent Blends). CL 202,347 has been reported also to be very soluble in ethyl acetate (AC 6747:15), but not quite soluble in hexane (limit to <1 mg/mL hexane).

PERSONNEL

A list of the personnel associated with this study is attached in Appendix C.

GLP COMPLIANCE

The statement of GLP Compliance, signed by the Study Director, is attached as Appendix D. The final report, raw data and a statement prepared and signed by ChemAlysis, Incorporated Quality Assurance Unit for this study is attached in ChemAlysis Report located in Appendix F.

STUDY INTEGRITY

There were no known circumstances that may have affected the quality or integrity of the data.

QUALITY ASSURANCE

A statement prepared and signed by the ChemAlysis, Incorporated Quality Assurance Unit for this study is attached in ChemAlysis Report (Appendix F). A statement prepared and signed by the Cyanamid Quality Assurance Unit covering the contents of this report is attached as Appendix E.

ARCHIVES

All raw data, protocol and amendments, and appropriate samples, documentation, records and the final report and amendments are stored in the Archives of the American Cyanamid Company, Agricultural Research Division, Princeton, New Jersey.

REFERENCES

ChemAlysis Report No. 910113

Sigit Witkonton 12/30/92

S. Witkonton
Study Director

Date

SW/ct

APPENDIX A

Method M 2243

M 2243
S. Witkonton/ct

Approved by:

J. Boyd 920914
J. Boyd Date

AMERICAN CYANAMID COMPANY
AGRICULTURAL RESEARCH DIVISION
HUMAN AND ENVIRONMENTAL SAFETY
P. O. Box 400
Princeton, New Jersey 08543-0400 USA

RECOMMENDED METHOD OF ANALYSIS

Herbicide, pendimethalin (CL 92,553): Determination of CL 92,553 and CL 202,347 (Metabolite) Residues in Canola Green Forage, Canola Seed and Canola Hay

A. Principle

CL 92,553 and CL 202,347 are extracted from canola seed with 1:3 isopropanol:hexane or from the other commodities with 1:1 methanol:water. Both compounds are partitioned into hexane. The hexane solution is cleaned up by passing an aliquot through a GPC column. The CL 92,553 and CL 202,347 eluates are cleaned up further with SPE LC-FLORISIL® using an ethyl acetate:hexane elution system. Both compounds are determined by fused silica capillary gas chromatography equipped with an electron capture detector. The analyses are accomplished using external standardization. A method sensitivity of 0.05 ppm is achieved for both compounds.

B. Apparatus (Items from other manufacturers may be used provided they are functionally equivalent).

1. Gas Chromatograph: An instrument equipped with an inlet system for a capillary column and with a standard Nickel-63 electron capture detector is suitable. The Tracor Model 565 gas chromatograph equipped with a packed column system can be adapted to fit the capillary column.

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2. Capillary Adaptors (if required): Adaptors for fused silica capillary column (0.53 mm nominal I.D.)

   a. Inlet: Adaptor with 1/4" O.D. x 90 mm long stem (SGE Part Number 103461O, Scientific Glass Engineering, Incorporated).

   b. Detector Connector: connector with make-up gas of 1/16" O.D. line (SGE Part Number 103462).

3. Fused Silica Capillary Column: Fused silica column, 15 M long x 0.53 mm nominal I.D. SPB-1 bonded phase with film thickness of 0.5μ (Supelco Cat. NO. 2-5314).

4. Ultrasonic Extractor: POLYTRON® Homogenizer, Model PT10ST (Brinkmann Instruments, Incorporated).

5. Filtration Apparatus: A 500-mL suction flask fitted with a 600-mL Buchner porcelain funnel by means of a rubber adapter.

6. Filter Paper: 9 cm (Whatman Number 40).

7. Rotary Evaporator: Buchler Flash-Evaporator (Buchler Instruments, Fort Lee, New Jersey), or equivalent, equipped with a water bath at about 40°C.

8. Evaporation Flasks: Round bottom, with TS 24/40 joint, 250-mL, 500-mL capacity and pear-shaped with TS 24/40 joint, 100-mL capacity (Kontes Glass Company, Vineland, New Jersey).

9. Solid Phase Extraction Cartridges: SPE LC-FLORISIL®, 1000 mg (6-mL) (Supelco Cat. No. 5-7057).

10. Laboratory Glassware: Assorted graduated cylinders, volumetric flasks, pipets and 250-mL separatory funnels.

11. GPC Column: 2.5 cm I.D. x 62 cm glass column (ABC Laboratories).

12. Metering Pump approximately 5 mL/min: Eldex Model No. E-120-S (Cat. No. ELD-1001).

13. Millex-SR Filter: (Millipore)

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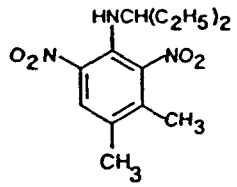
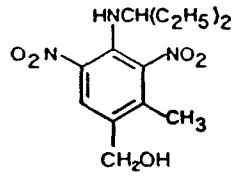
C. Reagents (Items from other manufacturers may be used provided they are functionally equivalent).

1. Analytical Standards: CL 92,553 and CL 202,347, analytical grade, known purity. Obtainable from American Cyanamid Company, Princeton, New Jersey 08540.

   a. CL 92,553: N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine



   b. CL 202,347: 4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol



2. Solvents: Residue analysis grade ("B & J" High Purity Solvent, American Burdick and Jackson, Muskegon, Michigan or equivalent).

   a. Cyclohexane

   b. Dichloromethane (methylene chloride)

   c. Ethyl Acetate

   d. Hexane

   e. Isopropanol

   f. Methanol

   g. Mixed Solvents - Measure the required volume of each component separately, combine and mix well.

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i. Extraction Solvent for Seed - 25% isopropanol in hexane.

ii. Extraction Solvent for Other Commodities - 50% methanol in water.

iii. GPC Solvent - 15% dichloromethane in cyclohexane.

iv. SPE Solvent 1 - 10% ethyl acetate in hexane.

v. SPE Solvent 2 - 20% ethyl acetate in hexane.

3. Bio-Beads: S-X3 (200/400 mesh), (Bio-Rad, Cat. No. 152-2750).

D. Preparation of Standard Solutions

Prepare separate standard solutions of CL 92,553 and CL 202,347 using the following procedures:

1. Stock Solutions

Accurately weigh about 100 milligrams (corrected for percent purity) of analytical standard into a small beaker. Dissolve the compound in ethyl acetate and quantitatively transfer the solution to a 100-mL volumetric flask. Dilute to the mark with ethyl acetate. This solution contains about 1.0 mg active ingredient/mL and is designated as the Standard Solution A from which other standard solutions may be prepared. This solution is stable for at least 5 months in room temperature if kept tightly stoppered.

2. Intermediate Standard Solution

Calculate the volume of Standard Solution A for both CL 92,553 and CL 202,347 which contains exactly 1.0 mg of the standard active ingredient and transfer that volume by pipet into a 100-mL volumetric flask; dilute to the mark with ethyl acetate. This solution contains 10 mcg/mL and is designated as the Standard Solution B.

3. Gas Chromatographic Working Standards

Pipet a 1-mL aliquot of Standard Solution B into a 10-mL volumetric flask and dilute to the mark with ethyl acetate. This solution contains 1 mcg standard/mL and is designated as Standard Solution C. Pipet a 5-mL aliquot of the Standard Solution C into a 10-mL volumetric flask and dilute to the mark with ethyl acetate. This solution contains 0.5 mcg standard/mL and is

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designated as Standard Solution D. Pipet a 5-mL aliquot of Standard Solution D into a 10-mL volumetric flask and dilute to the mark with ethyl acetate. This solution contains 0.25 mcg standard/mL and is designated as the Standard Solution E. Pipet a 5-mL aliquot of Standard Solution E into a 10-mL volumetric flask and dilute to the mark with ethyl acetate. This solution contains 0.125 mcg standard/mL and is designated as Standard Solution F. Pipet a 5-mL aliquot of Standard Solution F into a 10-mL volumetric flask and dilute to the mark with ethyl acetate. This solution contains 0.0625 mcg/mL and is designated as Standard Solution G. The working standards should be prepared fresh weekly to prevent solvent concentration.

4. Fortification and Linearity Standards

Utilize solutions for fortification of samples in the solutions. The concentration of fortification solutions 0.5-mL, 1-mL or 2-mL aliquot added to the same fortification level.

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Prepare standards for checking linearity of chro Standard Solution C (i.e., the 1 mcg/mL standa

E. Packing and Calibration of the Gel Permeation Chro

1. Packing

a. Weigh 50 grams of Bio-Beads S-X3 into a 2 150 mL of 15% dichloromethane in cyclohe soak overnight.

b. Set up the GPC column for upward flow (sample injector valve at the bottom end) of DCM-CH solvent at a rate of about 5 mL/minute driven by the Eldex metering pump.

c. Pour the Bio-Beads slurry into the column and drain the solvent; do not allow the bed to go dry at any time. Insert the upper piston into the column and adjust it so that its frit is just touching the top of the bed.

d. Start the metering pump and pump the DCM-CH solvent through the column continuously for about 2 days to firm up the bed. The solvent may be recycled in this operation.

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e. Carefully adjust the lower piston so that its frit is in contact with the lower end of the bed. This step should be repeated as necessary to be sure that there are no gaps between the bed and the frits at both ends.

2. Calibration

a. Mix CL 92,553 and CL 202,347 standard solutions containing 1 mg of each compound (1 mL of Solution A) in a evaporation flask. Evaporate to just dryness.

b. Dissolve the residue in 10 mL of the DCM-CH solvent and load into the 5-mL sample loop. Drain the excess into a beaker.

c. Place a 200-mL graduated cylinder below the column, start the flow of mobile phase and collect fractions.

d. Observe the movement of CL 92,553 and CL 202,347 bands by their yellowish color.

e. Cut the eluate fractions of both CL 92,553 and CL 202,347 in 100 mL graduate cylinders.

f. Evaporate the eluates to dryness in a 100-mL round bottom boiling flask.

g. Reconstitute both compounds in hexane using 100-mL volumetric flasks (expect 5 ng/µl)

h. Dilute the solution 1:5 in ethyl acetate (expect 1 ng/µl) for analysis.

i. For recovery assay, make up a final 1 ng/µl standard solution by pipetting Solution A, i.e., 1 mL of CL 92,553 and 1 mL of CL 202,347 into a 100-mL volumetric flask and dilute to 1:10 mL with ethyl acetate in a 10-mL volumetric flask.

j. Regenerate the GPC column by washing the column with 200 mL mobile phase.

k. Clean the sample loop thoroughly with DCM-CH.

l. Check for cross contamination by collecting 100 mL effluent and evaporate to dryness.

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m. Reconstitute the residue in 1 mL ethyl acetate and analyze by gas chromatography.

F. Gas Chromatographic Conditions

The operating conditions described below are provided for use as guides in establishing actual operating conditions and should be adjusted as necessary to obtain peak shape and resolution from background peaks equivalent to or better than those shown on the attached figures.

1. Oven Temperature: Programming

Initial Temperature	150°C	
Initial Time	2 min	
Program Rate	10°C/min	
Final Temperature	240°C	
Final Time	20 min	
Run Time	20 min	
Retention Time:	CL 92,553	3.0 min
	CL 202,347	4.7 min
Attenuation	64	

2. Inlet Temperature: 220°C

3. Injection Mode: On Column or Direct Injection

4. Carrier Gas Flow: 6 mL/min (5% methane in Argon).

5. Make-up Gas for EC Detector: 50 mL/min (5% methane in Argon).

G. Linearity Check

Check the instrument for linearity of chromatographic response every day it is used for the analysis of CL 92,553 and/or CL 202,347. Inject standard solutions of 0.0625, 0.125 and 0.25 mcg standard/mL (i.e., Standard Solutions G, F, E). Calculate the unit response (response/concentration) for each concentration and average the values. Departure at any concentration of more than 15% from the average indicates

instrument malfunction or faulty standard preparation which must be corrected before proceeding with sample analysis.

H. Recovery Test

The ability of the analyst to perform these procedures satisfactorily must be demonstrated by recovery tests before analysis of unknown samples is attempted. In addition, at least one recovery sample must be run with each batch of unknown samples.

Weigh a 10-g portion of untreated sample into a 250-mL Erlenmeyer flask and add by pipet the volume of a fortification solution to yield the desired fortification level. A practical guide is provided by the following Table:

Fortification Desired (ppm)	Standard Solution	Concentration of Standard Solution	Volume of Standard Solution Added
0.05	C	1.0 mcg/mL	0.5 mL
0.10	C	1.0 mcg/mL	1.0 mL
0.50	B	10.0 mcg/mL	0.5 mL

I. Sample Handling Procedure

Freeze a quantity of the commodity sufficient to provide representative sampling, mix with powdered dry ice, and chop with a pre-chilled food chopper. Store the sample in a freezer until the dry ice has dissipated.

1. Extraction

a. Canola Green Forage and Canola Hay

Weigh a 10.0-gram portion of the sample into a 250-mL Erlenmeyer flask. Add 200 mL of 50% methanol in water and allow to stand for about 17 hours (overnight). Blend the mixture with a POLYTRON® homogenizer for about 2 minutes. Place a Whatman No. 40 filter paper in a Buchner funnel and wash with 50 mL of methanol. Discard the methanol wash and filter the homogenate with vacuum into a 500-mL filtering flask. Return the filter cake to the blending flask, add 200 mL of methanol and blend the mixture again for about 2 minutes. Filter the second homogenate through the same filter paper. Transfer the filtrate to a 500-mL stoppered

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graduated cylinder. Adjust the volume to 400 mL with methanol. Stopper the cylinder and shake to mix the contents thoroughly. Transfer a 100-mL aliquot (2.5-gram sample size equivalent) to a 500-mL evaporation flask and concentrate the solution to about 25 mL.

b. Canola Seed

Weigh a 10.0-gram portion of the sample into a 250-mL Erlenmeyer flask. Add 200 mL of 25% isopropanol in hexane and blend the mixture with a POLYTRON® homogenizer for about 2 minutes. Place a Whatman No. 40 filter paper in a Buchner funnel and wash with 50 mL of extracting solvent.

Discard the solvent wash and filter the homogenate with vacuum into a 500-mL filtering flask. Return the filter cake to the blending flask, add 200 mL of 25% isopropanol in hexane and blend the mixture again for about 2 minutes. Filter the second homogenate through the same filter paper. Transfer the filtrate to a 500-mL stoppered graduated cylinder. Adjust the volume to 400-mL with 25% isopropanol in hexane. Stopper the cylinder and shake to mix the contents thoroughly. Transfer a 100-mL aliquot (2.5-gram sample size equivalent) to a 500-mL evaporation flask and evaporate to dryness.

2. Partition

a. Canola Green Forage and Canola Hay

Transfer the solution to a 250-mL separatory funnel. Add 50 mL of hexane to the evaporating flask and swirl. Transfer the hexane to the separatory funnel. Shake the contents in the separatory funnel vigorously for 40 seconds. Draw off the bottom (aqueous) layer into the original evaporating flask and transfer the top (hexane) layer to a clean 250-mL evaporation flask. Repeat the extraction of the aqueous layer twice more with 50-mL portions of hexane. Evaporate the combined hexane to dryness on the rotary evaporator.

b. Canola Seed

Wash the residue in the 500-mL evaporating flask successively with 50 mL hexane, 25 mL distilled water and transfer each solution to a 250-mL separatory funnel. Shake the contents in the separatory funnel vigorously for 40 seconds. Draw off the bottom (aqueous) layer into the original evaporating flask and transfer the top (hexane) layer to a clean 250-mL

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evaporation flask. Return the aqueous extract to the 250-mL separatory funnel. Wash the 500-mL evaporating flask again with 50 mL hexane and decant into the 250-mL separatory funnel. Shake the mixture vigorously for 40 seconds. Draw off the bottom layer into the 500-mL evaporating flask and transfer the hexane layer to combine with the first extract in the 250-mL evaporation flask. Repeat the above procedure one more time to complete the partition step.

3. Cleanup

a. Gel Permeation Chromatography (GPC)

Conditions:

Column: 2.5 cm I.D. x 62 cm glass column, packed with 50 g Bio-Beads S-X3 (Bio Rad) (200/400 mesh) to a bed length of 29 cm

Solvent System: Dichloromethane-cyclohexane 15:85 (DCM-CH)

Flow Rate: 5.0 mL/min (after firmly packed)

Dump Volume: First 130 mL (approximate) (Macromolecules such as chlorophyll and lipids)

CL 92,553 Elution Volume: 131-160 mL (approximate)

CL 202,347 Elution Volume: 216-269 mL (approximate)

Wash Volume: 100 mL

Procedure:

1) Load the canola extract residue (i.e., after the hexane extract was evaporated to dryness) by dissolving the residue in 10 mL of DCM-CH and load into the 5 mL loop. (Use Millex-SR filter if sample has particulates).

2) Elute the column with DCM-CH solvent.

3) Collect the first eluate (1-130 mL) and discard.

4) Collect the CL 92,553 eluate (131-160 mL).

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5) Collect the CL 202,347 eluate (216-269 mL).

6) Regenerate the column with 100 mL of DCM-CH solvent.

7) Load the next sample residue.

b. SPE LC-Florisil Cartridge Clean-up:

CL 92,553

1) Evaporate the GPC eluate just to dryness.

2) Wash the cartridge with 5 mL hexane.

3) Load the sample residue with 3 x 2 mL hexane.

4) Discard the hexane eluate.

5) Elute CL 92,553 with 5 mL of 10% ethyl acetate in hexane.

6) Evaporate to dryness.

7) Dissolve in 1 mL ethyl acetate for GC analysis.

CL 202,347

1) Evaporate the GPC eluate just to dryness.

2) Wash the cartridge with 5 mL 10% ethyl acetate in hexane.

3) Load the sample residue with 3 x 2 mL 10% ethyl acetate in hexane.

4) Discard the 10% ethyl acetate in hexane eluate.

5) Elute CL 202,347 with 10 mL of 20% ethyl acetate in hexane.

6) Evaporate to dryness.

7) Dissolve in 1 mL ethyl acetate for GC analysis.

J. Gas Chromatography

Inject standard and sample solutions alternately as follows: standard, sample in duplicate, standard, sample in duplicate, etc., allowing late-eluting peaks to clear between each injection. If the sample response exceeds full-scale pen deflection at the sensitivity used, make proper dilutions of the sample solution to keep the response on scale and record the dilution factor. Measure peak heights with a millimeter ruler or with an electronic integrator. If the duplicate injections for a given sample differ by more than 15% reinject the "standard, sample, sample, standard" set. In similar fashion, if the preceding and following standards, do not agree to within 15%, reinject the set.

K. Calculations

For purposes of calculations, use the average peak height of the duplicate injections for each samples and the average peak height for the standard immediately preceding and immediately following that sample. Calculate the apparent residues as follows:

$$\text{Apparent residues (ppm)} = \frac{R(SAMP) * V1 * V3 * V5 * C(STD) * DF}{R(STD) * W * V2 * V4}$$

Where:

R(SAMP) = Average sample response (mm or integrator units)

R(STD) = Average standard response (mm or integrator units)

C(STD) = Concentration of Working Standard (mcg/mL)

V1 = Initial volume of sample extract (400 mL)

V2 = Volume of equivalent sample aliquot used for analysis (50 mL)

V3 = Final volume of sample solution for GLC (1.0 mL)

V4 = Volume of sample solution injected (1.0 mcL)

V5 = Volume of standard solution injected (1.0 mcL)

W = Sample weight (10 grams)

DF = Dilution Factor (1)

(Values in parentheses are nominal values if the procedure is carried out exactly as described.)

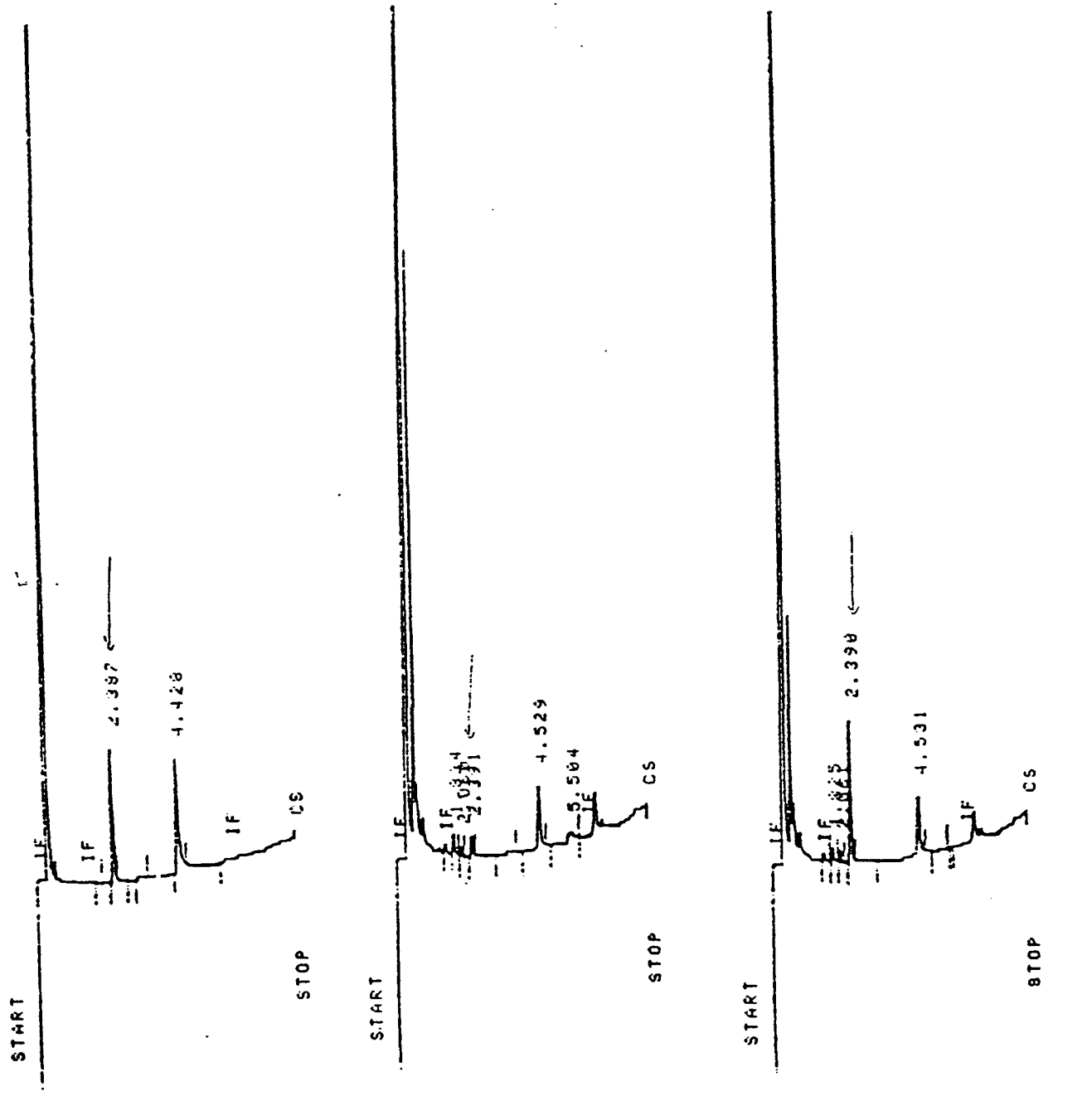
L. Notes on the Procedure

1. All elution operations with SPE cartridges are accomplished by gravity, i.e., no vacuum or pressure used.

2. Before use, each lot of SPE LC-FLORISIL® cartridges must be checked for potential interferences. Prior to use, elute the SPE LC-FLORISIL® cartridge with 5 mL of ethyl acetate, evaporate the ethyl acetate and analyze for interference; if interferences are observed, notify the manufacturer (Supelco guarantee for replacement). SPE LC-FLORISIL® cartridges showing minor interferences may be used, but must be cleaned before use by washing them with 5 mL ethyl acetate followed by 5 mL hexane. The sample may then be loaded immediately.

3. Hexane may be used as an alternate gas chromatography injection solvent for standards and sample extracts provided that all the criteria set in the method can be met.

Figure 1: Gas Chromatograms of Extracts of Control and Fortified Canola Hay Analyzed for CL 92,553



CL 92,553 Standard, 0.0625 mcg/mL (1 mcL)

Canola Hay Control, CL 92,553 Dilution Factor = 1

Canola Hay, Fortified at 0.05 ppm with CL 92,553 Dilution Factor = 1

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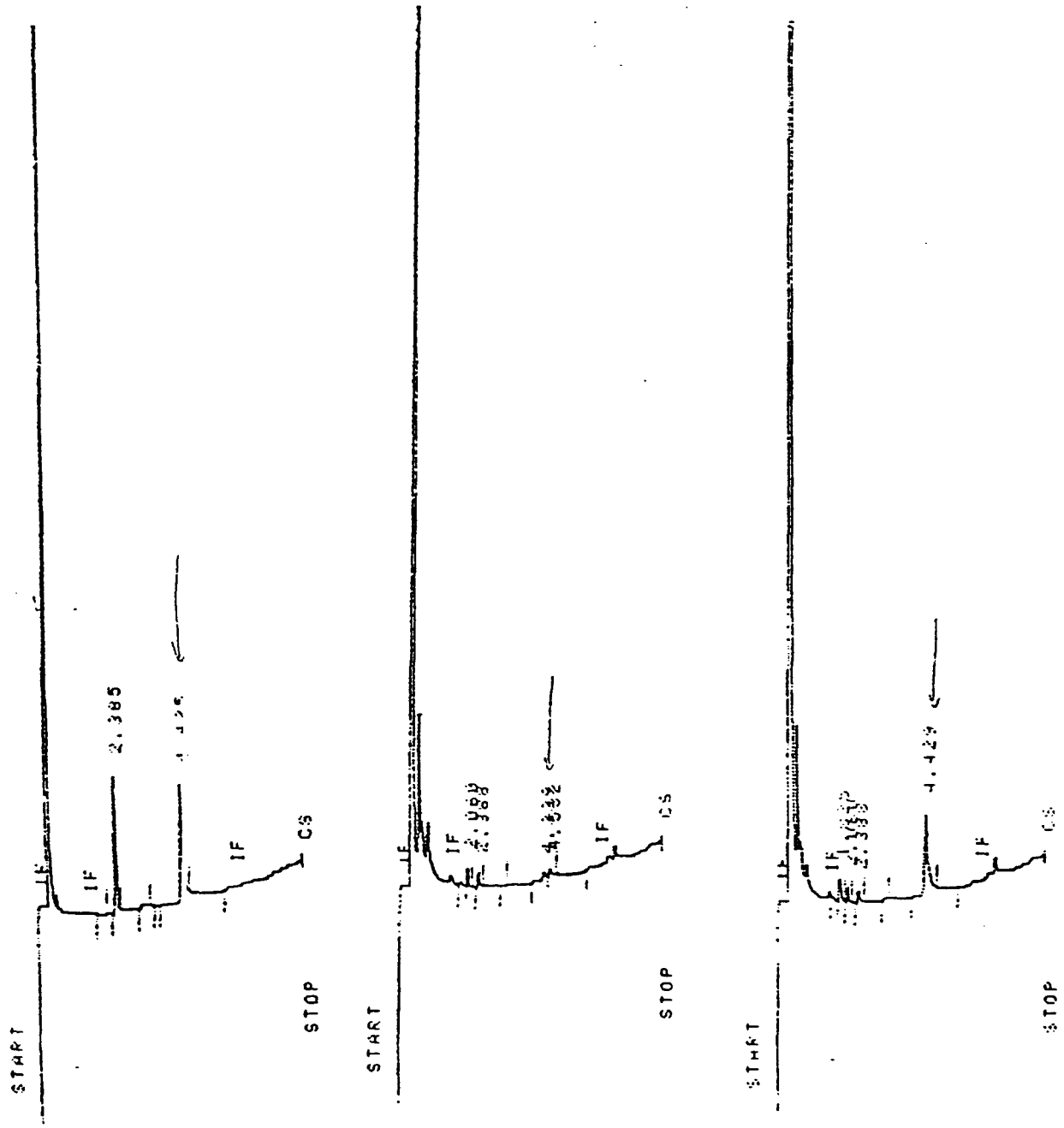
Figure 2: Gas Chromatograms of Extracts of Control and Fortified Canola Hay Analyzed for CL 202,347



CL 202,347 Standard, 0.0625 mcg/mL (1 mcL)

Canola Hay Control, CL 202,347
Dilution Factor = 1

Canola Hay, Fortified at 0.05 ppm with CL 202,347
Dilution Factor = 1

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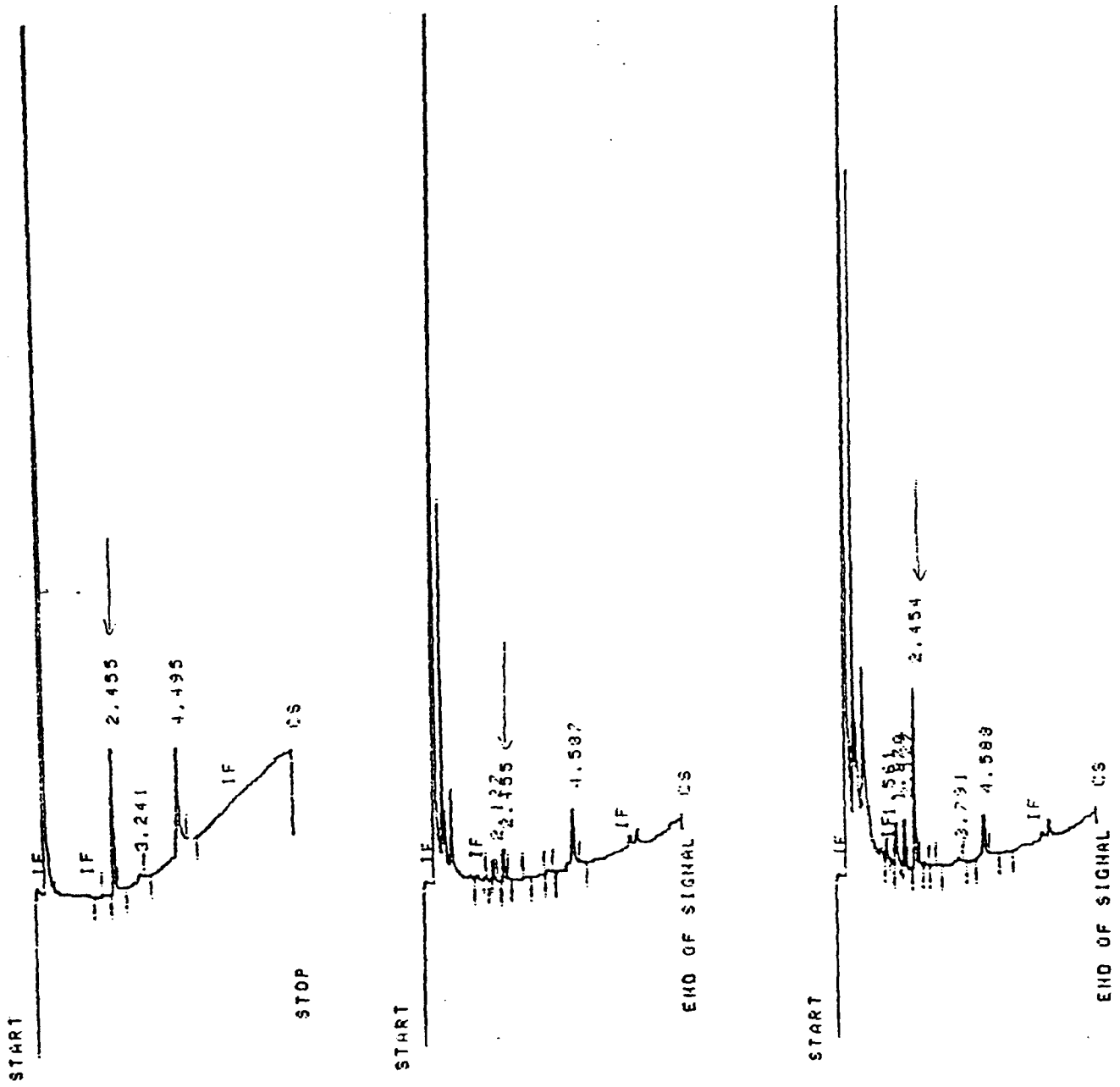
Figure 3: Gas Chromatograms of Extracts of Control and Fortified Canola Green Forage Analyzed for CL 92,553

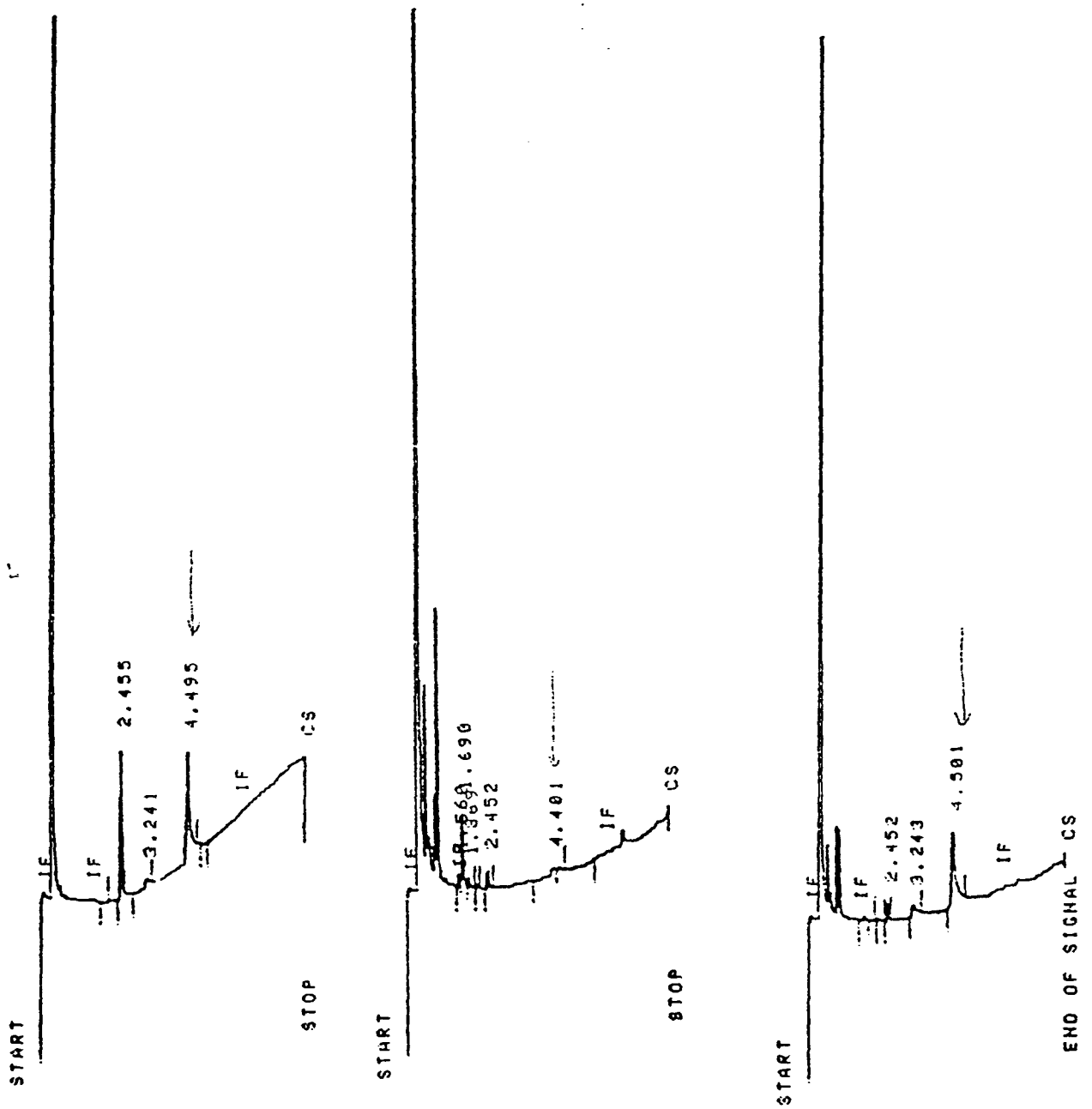


CL 92,553 Standard, 0.0625 mcg/mL (1 mcL)

Canola Green Forage Control CL 92,553 Dilution Factor = 1

Canola Green Forage, Fortified at 0.05 ppm with CL 92,553 Dilution Factor = 1

Figure 4: Gas Chromatograms of Extracts of Control and Fortified Canola Green Forage Analyzed for CL 202,347

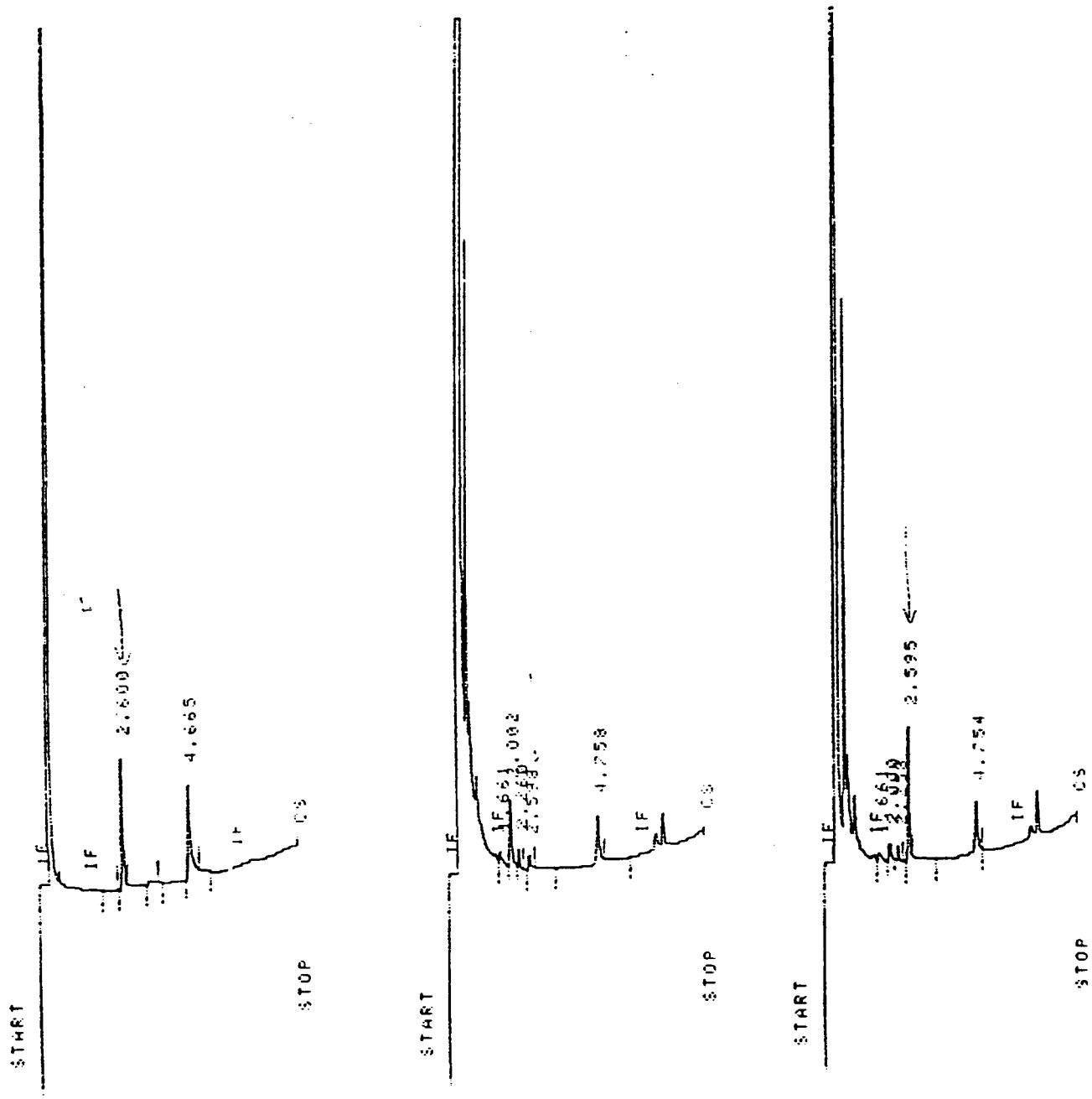


CL 202,347 Standard, 0.0625 mcg/mL (1 mcL)

Canola Green Forage Control CL 202,347 Dilution Factor = 1

Canola Green Forage, Fortified at 0.05 ppm with CL 202,347 Dilution Factor = 1

Figure 5: Gas Chromatograms of Extracts of Control and Fortified Canola Seed Analyzed for CL 92,553

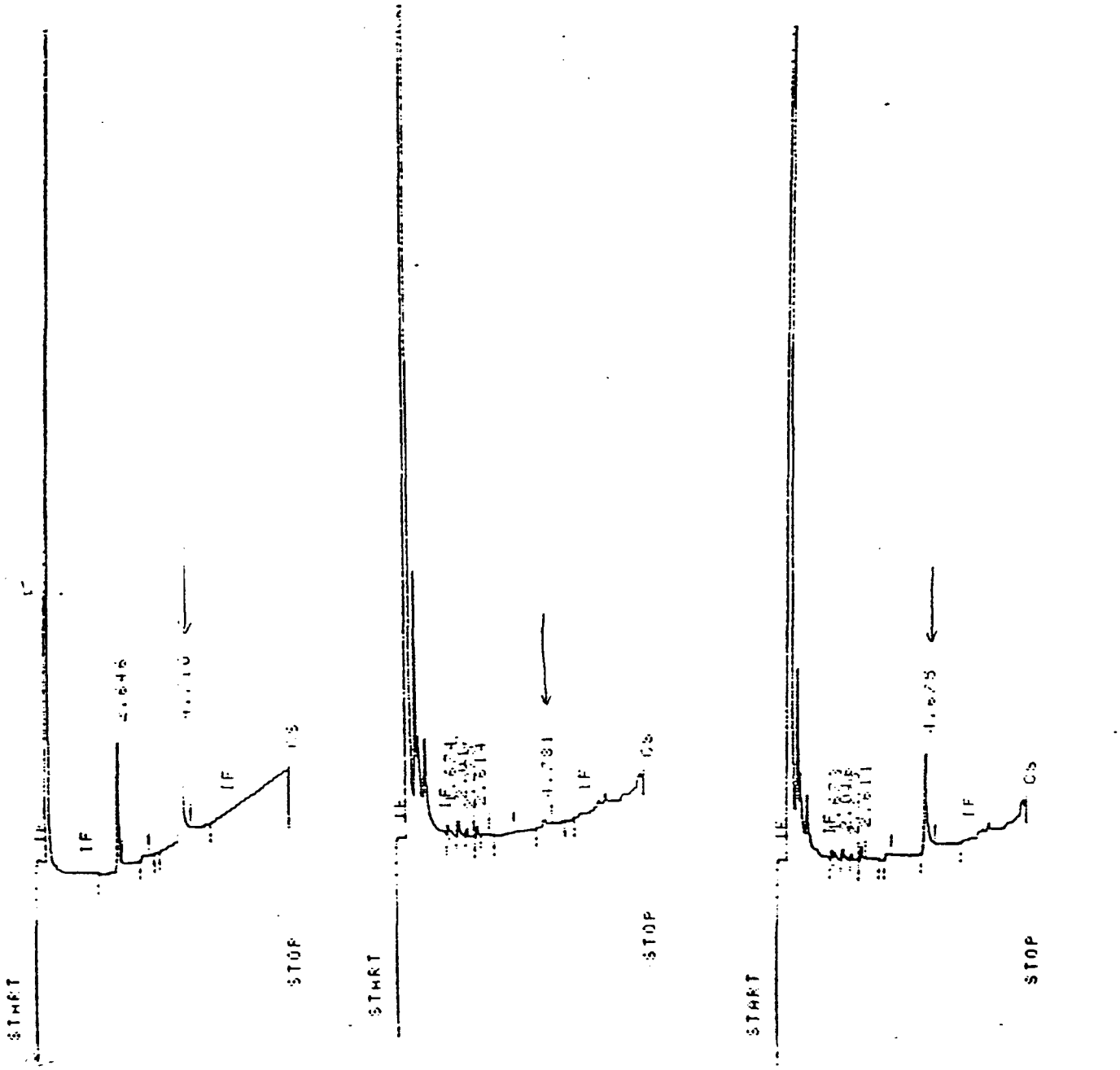


CL 92,553 Standard, 0.0625 mcg/mL (1 mcL)

Canola Seed Control, CL 92,553 Dilution Factor = 1

Canola Seed, Fortified at 0.05 ppm with CL 92,553 Dilution Factor = 1

Figure 6: GC as Chromatograms of Extracts of Control and Fortified Canola Seed Analyzed for CL 202,347



CL 202,347 Standard, 0.0625 mcg/mL (1 mcL)

Canola Seed Control, CL 202,347 Dilution Factor = 1

Canola Seed, Fortified at 0.05 ppm with CL 202,347 Dilution Factor = 1

APPENDIX B

**Residue Support Study Protocol,
Amendment and Deviation**

PR92PT01

RESIDUE SUPPORT STUDY PROTOCOL

Distribution:
0941 File
All those signing
Cyanamid Quality Assurance
D. Barringer

Protocol No : PR92PT01

Title:

Validation of Method M 1930.01 for the Determination of CL 92,553 and CL 202,347 residues in Canola Forage, Hay, and Seed at Chemalysis, Inc.

Purpose:

To conduct a laboratory validation of Method M 1930.01 for the determination of CL 92,553 and CL 202,347 residues in Canola Forage, Hay, and Seed.

Sponsor/Test Facility:

American Cyanamid Company
Agricultural Research Division
P.O. Box 400
Princeton, NJ 08543-0400

Test Site:

ChemAlysis, Inc.
P.O. Box 1010
8510 Corridor Road
Savage, MD 20763

Test Material:

1. Analytical Standard of CL 92,553 (Lot AC 5105-7; Purity of 98.1%; Expiration Date is 7/93).

2. Analytical Standard of CL 202,347 (Lot AC 6127-126; Purity of 97%; Expiration Date is 8/93).

These standards will be used for the fortification of canola forage, hay, and seed as well as the external standards in the analyses.

Data regarding the characterization of the test material reside in the archives of the Analytical, Physical and Biochemical Section of American Cyanamid Company.

Test System:

1. Sample Type: Untreated canola forage, hay, and seed.

2. Source:

   Canola Seed: AC 6105.50
   Canola Forage: AC 6794.81A
   Canola Hay: AC 6794.81B

3. Justification:

   The canola forage, hay, and seed selected are representative of commodities collected from field experiments and processed for residue analysis. They are appropriate for demonstrating the validity of this analytical method after fortification with known levels of the analytes.

4. Identification of the Test System:

   Identify each sample in the laboratory, in the raw data and in the report with a unique number consisting of the sample number for commodity, the fortification level and the replicate number. For example:

   Each of the duplicate analyses at 0 ppm of canola forage, AC 6794.81A, would be identified as:

   6794.81A-1 and 6794.81A-2.

   Each of the duplicate analyses at 0.050 ppm of canola hay, AC 6794.81B, would be identified as:

   6794.81B-0.050-1 and 6794.81B-0.050-2.

Personnel:

1.	Study Director:	Sujit Witkonton American Cyanamid Company
2.	Principal Analyst:	Tarun Mehta ChemAlysis
3.	Quality Assurance:	ChemAlysis QA Personnel

Method Number: Cyanamid Method M 1930.01

Time Frame

Proposed Experiment Start Date: January 20, 1992
Proposed Experiment Completion Date: February 20, 1992

Experimental Design:

1. Add known amounts of CL 92,553 and CL 202,347 standard solutions to control canola samples to give the desired fortification levels.

2. Follow Method M1930.01 as written for the analysis of the fortified canola forage, hay, and seed samples.

3. Typical recoveries must fall between 70% and 120%. If there is a problem meeting this requirement, notify the Study Director, who will be able to provide assistance.

4. When a recovery does not fall within the accepted range (70-120%) the recovery will be repeated an additional two times and all recovery values averaged for the report.

5. Perform recoveries in duplicate on the control canola forage, hay, and seed samples covering the following fortification levels:

0.00	ppm
0.05	ppm
0.10	ppm
0.50	ppm

Handling of Data and Records to be Maintained:

The following records should be maintained for this study:

1. Complete sample history, including storage conditions, from the time of arrival at ChemAlysis Labs, Inc. until sample is completely used or returned to Cyanamid.

2. History of standards, preparation of stock solutions, records of standard dilutions and preparation of final working standards.

3. Any modifications to the method as described in the protocol.

4. Calculations for the determination of peak heights and conversion to ppm of all chromatography.

5. All chromatography including standards, controls, treated samples and concurrent recoveries along with appropriate sample tracking and injection schedules.

6. All personnel involved in the study.

7. Instrument conditions of each analytical run.

8. Statement of Compliance to GLP.

9. Quality Assurance Statement including phase and data audited.

Statistical Methods:

Determine the mean of the recoveries for each commodity at each fortification level, and determine the mean and standard deviation of all the recoveries for each commodity.

Reporting of Results:

Write a validation report that presents the results from this study. Show recovery values, the mean of the recoveries for the duplicate samples, and the mean and standard deviation of all the recoveries for each commodity.

Protocol Amendments:

All changes in or revisions of the approved protocol and the reasons for the changes will be documented, signed by the Study Director, dated and maintained with the protocol.

Quality Assurance:

The study will be performed in compliance with Good Laboratory Practice Standards as specified in 40 CFR 160. A statement of compliance or noncompliance with Good Laboratory Practices will be signed by the Study Director and the Principal Analyst at the conclusion of the study.

Approvals:

Study Director: Sujit Witkonton 1/15/92
Sujit Witkonton Date

Principal Analyst (ChemAlysis): Tarun Mehta 1/14/92
Tarun Mehta Date EE 82 1/15/92

Group Leader:
Residue Chemistry I John Boyd 920115
John Boyd Date

RESIDUE SUPPORT STUDY PROTOCOL AMENDMENT

PROTOCOL NUMBER: PR92PT01

Copy To: MREE File
Cyanamid Quality Assurance
MREE Chemical Archivist
Manager, Residue Chemistry
Those Signing Protocol
Laboratory Personnel

AMENDMENT NUMBER: 01

STUDY TITLE: Validation of Method M 1930.01 for the Determination of CL 92,553 and CL 202,347 Residues in Canola Forage, Hay and Seed at ChemAlysis, Inc.

AMENDMENT(S) TO BE MADE:

The study title will be change to:

Validation of Method M 2243 for the Determination of CL 92,553 and CL 202,347 Residues in Canola Forage, Hay and Seed at ChemAlysis, Inc.

REASON(S) FOR THE AMENDMENTS:

To correct study title. GC method M 1930.01 was used as a preliminary method to validate GC method M 2243.

IMPACT ON STUDY:

None

APPROVALS:

Sujit Witkonton — 9/11/92
Sujit Witkonton
Study Director
Date

John E. Boyd — 920914
John E. Boyd
Group Leader
Residue Chemistry I
Date

JB/ct

CYANAMID

Res. 111.00 9/91

PROTOCOL DEVIATION

Deviation Number: 1 **Protocol #:** CL92PT01

Study Title: Validation of Method M2243 for the Determination of CL92553 and CL202,347 Residues in Canola Forage, Hay and Seed at ChemAlysis Inc

Description of Deviation:

1. The purity of test materials in the protocol were different from the values in the ChemAlysis report.

2. Values of 183% (Run #755, Table 2B) and 230% (Run #853, Table 3B), were not included in Average Calculation.

3. The 121% sample (Table 2B, 6794.81A-ovs-1) was not repeated

Reason for Deviation: 1. The purity in the protocol are the latest re-assayed values. 2. Both values were suspicious of fortification errors (double fortifications)

3. This sample was not repeated due to being insignificant from the upper limit of 120% recovery

Impact on Study: None (except #2 may make the average recovery look better than it should if included 183% and 230% in the calculation

Signature: Sujit Velkanta **Date:** 11/30/92

Study Director's Approval:

Signature: Sujit Velkanta **Date:** 11/30/92

Distribution:

Study File (original)
QA Coordinator Dept. 0941 (copy)
Those signing (copy)
ACCO QAU (copy)
Lab. Personnel involved (copy)

APPENDIX C

Personnel

STUDY PERSONNEL

NAME	TITLE	COMPANY
Sujit Wit conton	Study Director	American Cyanamid Company
John Boyd	Group Leader, Residue Group (I)	American Cyanamid Company
Tarun D. Mehta	Principal Analyst	ChemAlysis, Inc.
Violeta Burgos	Chemist	ChemAlysis, Inc.
Julie Moyer	Lab Technician	ChemAlysis, Inc.
Lorraine Lovett	Acting Quality Assurance Director	ChemAlysis, Inc.

APPENDIX D

Statement of GLP Compliance

American Cyanamid Company
Agricultural Research Division
P. O. Box 400
Princeton, New Jersey 08543-0400
(609) 799-0400

Human and Environmental Chemistry
Metabolism, Residue and Environmental Chemistry and Ecotoxicology

Report Number: C 3961

Study Number: PR92PT01

STATEMENT OF GLP COMPLIANCE

This study was conducted in accordance with Good Laboratory Practice regulation 40 CFR part 160 established by the Federal Insecticide, Fungicide and Rodenticide Act.

S. Witkonton — 12/30/92

S. Witkonton
Study Director

Date

APPENDIX E

Quality Assurance Statement

CYANAMID

Confidential

QUALITY ASSURANCE STATEMENT

STUDY PR92PT01

REPORT NUMBER: C3961

The Quality Assurance Unit, Agricultural Research Division, American Cyanamid Company, conducted the following inspections/ audits for this study.

Phase	Date Performed	Date Reported
PROTOCOL REVIEW	20-JAN-92	20-JAN-92
REPORT REVIEW	02-OCT-92	02-OCT-92

Steven M. Boege
Quality Assurance Specialist

12/23/92
Date

APPENDIX F

ChemAlysis Final Report

Analytical Report Title

Validation of Method M1930.01
for the Determination of Pendimethalin (CL 92,553) and
Metabclite (CL 202,347) Residues in Canola Green Forage,
Hay, and Seed at ChemAlysis, Inc.

Client

American Cyanamid Company
Agricultural Research Division
P.O. Box 400
Princeton, New Jersey 08543-0400
(609) 799-0400

Study Director

Sujit Witkonton, Ph.D.
American Cyanamid Company

Performing Laboratory

ChemAlysis, Inc.
8510 Corridor Rd.
Savage, Maryland 20763
(301) 776-8388

Protocol No.

PR92PT01

Laboratory Project No.

910113

Analytical Initiation Date: 02/20/92
Analytical Completion Date: 03/06/92

Table of Contents

Good Laboratory Practice Statement

This study "Validation of Method M1930.01 for the Determination of Pendimethalin (CL 92,553) and Metabolite (CL 202,347) Residues in Canola Green forage, Hay, and Seed" for Protocol PR92PT01 was conducted according to EPA Good Laboratory Practice Standards, 40 CFR 160.

Sujit Witkonton, Ph.D.
Study Director

8/13/92
Date

Tarun D. Mehta
Principal Analyst

3/16/92
Date

Quality Assurance Monitoring Statement

Validation of Method M1930.01 for the determination of CL 92,553 and CL 202,347 residues in Canola Forage, Hay, and Seed at ChemAlysis, Inc.

The ChemAlysis portion of this study has been audited in accordance with Good Laboratory Practice Standards 40 CFR 160, specific protocols and ChemAlysis SOPs, and found to be in compliance by the ChemAlysis Quality Assurance Unit.

The following specifies the dates inspections were made and findings reported to the study director and to the study director's management.

Type of Audit	Phase Audited	Audit Date	Reported to Study Director	Reported to Management
Data	Raw data package (chromatograms, notebook pages, spreadsheets)	02/25/92	03/17/92	03/17/92
Physical Inspection	Extraction and Partition	03/03/92	03/17/92	03/17/92
Data	Raw data package	03/10/92	03/17/92	03/17/92
Data	Raw data package	03/12/92	03/17/92	03/17/92
Report	Final Report	03/17/92	--	--

Lorraine C. Lovett
Lorraine C. Lovett
Acting Quality Assurance Director

18 March 92
Date

Project Personnel

The Principal Analyst for "Validation of Method M1930.01 for the Determination of CL 92,553 and CL 202,347 residues in Canola Forage, Hay, and Seed at ChemAlysis, Inc.", Protocol No. PR92PT01, was Tarun D. Mehta, Laboratory Supervisor at ChemAlysis, Inc.

Supervising the conduct of the study for American Cyanamid Co. was Sujit Witkonton, Ph.D., Study Director.

The following ChemAlysis, Inc. personnel were associated with various aspects of the study:

Name	Title	Signature	Initials
Anthony F. Grigor	Technical Director	Anthony F. Grigor	AFG
Tarun D. Mehta	Lab Supervisor	Tarun Mehta	TM
Violeta Burgos	Chemist	Violeta Burgos	VB
Julie Moyer	Lab Technician	Julie Moyer	JM
Lorraine Lovett	Acting QA Director	Lorraine Lovett	LL
Byron Leo	QA Coordinator	Byron Leo	B.L
Scott Snouffer	Sample Custodian	(On file)	

Introduction

The purpose of this study is to conduct a laboratory validation of Method M1930.01 for the determination of Pendimethalin (CL 92,553) and metabolite (CL 202,347) residues in the commodities of Canola Green Forage, Hay, and Seed.

Materials

A. Analytical Standards

Analytical standards were supplied by American Cyanamid Co. and received at ChemAlysis, Inc. on 12/20/90 (page 12-13 Raw Data Package). The standards are summarized below:

Compound	Reference No.	Purity	Expiration Date
CL 92,553	AC5105-7	97.5%	07/93
CL 202,347	AC6127-126	90.0%	08/93

The analytical standards were stored in a freezer at <-10°C. Prepared GC standards and spiking solutions were kept refrigerated at 4°C ± 2°C. Certificates of analysis are maintained by the sponsor.

B. Samples

Samples of canola green forage, hay, and seed were received frozen by ChemAlysis, Inc. from American Cyanamid Co. on 1/7/92 (page 15 Raw Data Package). Samples are kept in a freezer at <-10°C when not in use.

Sample identification per protocol.

C. Apparatus

A description of apparatus used is included at the end of this report (page 17 Raw Data Package).

Methods

A. Residue Method

Samples were extracted and analyzed according to American Cyanamid Co. Method M1930.01 which is included at the end of this report (page 17 Raw Data Package). Samples were injected in an HP5890A gas chromatograph equipped with a electron capture detector, an HP7673A auto-injector and an HP3396A integrator.

B. Calculation

$$ppm = \frac{AVG\ R(SAMP) \times (V1) \times (V3) \times (V5) \times C(STD) \times DF}{AVG\ R(STD) \times (W) \times (V2) \times (V4)}$$

See page 28 in the Raw Data Package for value descriptions.

C. Statistical Method

Determine the mean for each commodity at each fortification level, and determine the mean and standard deviation of all the recoveries. (Table 4).

D. Final Report

The original final report and raw data will be archived at American Cyanamid Company, Agricultural Research Division, P.O. Box 400, Princeton, New Jersey 08543-0400. True and certified copies will be archived at ChemAlysis, Inc., Savage, Maryland 20763.

Conclusion

The validation study results for analysis of canola green forage, hay, and seed reveal that analytical Method M1930.01 is valid to determine Pendimethalin (CL 92,553) and metabolite (CL 202,347) residues in matrix field samples.

List of Tables

TABLE 1A: DETAILED RECOVERY DATA FOR METHOD VALIDATION OF CL 92,553
IN CANOLA SEED

COMMODITY: CANOLA SEED
PRODUCT: CL 92,553
ANALYTE: CL 92,553

METHOD USED: M1930.01
RESPONSE MEASURED AS: INTEGRATOR UNITS (HEIGHT)*
CHEMALYSIS PROJECT NO.: 910113

$$PPM = \frac{AVG\ R(SAMP) \times (V1) \times (V3) \times (V5) \times C(STD) \times DF}{AVG\ R(STD) \times (W) \times (V2) \times (V4)}$$

$$\%RECOVERY = \frac{(PPM\ FOUND\ IN\ FORTIFIED\ CONTROL - AVG\ PPM\ FOUND\ IN\ CONTROL) \times 100}{PPM\ ADDED}$$

NOTE: V4 = V5 = 1ul, thus cancel out of the ppm equation.
**Client ID Control AC 6105.50

SAMPLE IDENTIFICATION					FORTIFICATION				SAMPLE PREPARATION					SAMPLE ANALYSIS AND RESULTS					
SAMPLE IDENTIFICATION**		RUN NUMBER	DATE EXTRACTED	DATE ANALYZED	NOTEBOOK #118	FV (ml)	FC ug/ml	PPM ADDED	W (gm)	V1 (ml)	V2 (ml)	V3 (ml)	DIL. FAC.	C(STD) (ug/ml)	* R(SAMP)	AVG R(SAMP)	* AVG R(STD)	APPARENT PPM FOUND	PERCENT RECOVERY
6105.50-3	(1)	1003	3-9-92	3-11-92	pg. 27	--	--	--	10	400	50	1.0	1	0.0625	1201				
6105.50-3	(2)	1004	3-9-92	3-11-92	pg. 27	--	--	--	10	400	50	1.0	1	0.0625	1205	1203	9022	0.007	
6105.50-4	(1)	1005	3-9-92	3-11-92	pg. 27	--	--	--	10	400	50	1.0	1	0.0625	1095				
6105.50-4	(2)	1006	3-9-92	3-11-92	pg. 27	--	--	--	10	400	50	1.0	1	0.0625	1058	1077	9022	0.006	
6105.50-0.05-3	(1)	1009	3-9-92	3-11-92	pg. 27	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	8698				
6105.50-0.05-3	(2)	1010	3-9-92	3-11-92	pg. 27	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	8751	8725	9041	0.05	96
6105.50-0.05-4	(1)	1011	3-9-92	3-11-92	pg. 27	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	8971				
6105.50-0.05-4	(2)	1012	3-9-92	3-11-92	pg. 27	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	9017	8994	9041	0.05	99
6105.50-0.10-3	(1)	1015	3-9-92	3-11-92	pg. 27	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	16069				
6105.50-0.10-3	(2)	1016	3-9-92	3-11-92	pg. 27	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	15935	16002	9182	0.09	87
6105.50-0.10-4	(1)	1017	3-9-92	3-11-92	pg. 27	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	16424				
6105.50-0.10-4	(2)	1018	3-9-92	3-11-92	pg. 27	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	16783	16604	9182	0.09	90
6105.50-0.50-3	(1)	1021	3-9-92	3-11-92	pg. 27	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	17084				
6105.50-0.50-3	(2)	1022	3-9-92	3-11-92	pg. 27	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	17094	17089	9067	0.47	94
6105.50-0.50-4	(1)	1023	3-9-92	3-11-92	pg. 27	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	17331				
6105.50-0.50-4	(2)	1024	3-9-92	3-11-92	pg. 27	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	17662	17497	9067	0.48	96

CALCULATIONS PERFORMED BY: Violeta Bueso

CALCULATIONS CHECKED BY: Tarun Mehta

TABLE 1B: DETAILED RECOVERY DATA FOR METHOD VALIDATION OF CL 202,347 IN CANOLA SEED

COMMODITY: CANOLA SEED
PRODUCT: CL 92,553
ANALYTE: CL 202,347

METHOD USED: M1930.01
RESPONSE MEASURED AS: INTEGRATOR UNITS (HEIGHT)*
CHEMALYSIS PROJECT NO.: 910113

$$PPM = \frac{AVG\ R(SAMP) \times (V1) \times (V3) \times (V5) \times C(STD) \times DF}{AVG\ R(STD) \times (W) \times (V2) \times (V4)}$$

$$\%RECOVERY = \frac{(PPM\ FOUND\ IN\ FORTIFIED\ CONTROL - AVG\ PPM\ FOUND\ IN\ CONTROL) \times 100}{PPM\ ADDED}$$

NOTE: V4 = V5 = 1ul, thus cancel out of the ppm equation.
**Client ID Control AC 6105.50

SAMPLE IDENTIFICATION					FORTIFICATION				SAMPLE PREPARATION					SAMPLE ANALYSIS AND RESULTS					
SAMPLE IDENTIFICATION**		RUN NUMBER	DATE EXTRACTED	DATE ANALYZED	NOTEBOOK #118	FV (ml)	FC ug/ml	PPM ADDED	W (gm)	V1 (ml)	V2 (ml)	V3 (ml)	DIL. FAC.	C(STD) (ug/ml)	* R(SAMP)	AVG R(SAMP)	* AVG R(STD)	APPARENT PPM FOUND	PERCENT RECOVERY
6105.50-3	(1)	979	3-9-92	3-10-92	pg. 27	--	--	--	10	400	50	1.0	1	0.0625	<500				
6105.50-3	(2)	980	3-9-92	3-10-92	pg. 27	--	--	--	10	400	50	1.0	1	0.0625	<500	<500	6033	<0.005	
6105.50-4	(1)	981	3-9-92	3-10-92	pg. 27	--	--	--	10	400	50	1.0	1	0.0625	<500				
6105.50-4	(2)	982	3-9-92	3-10-92	pg. 27	--	--	--	10	400	50	1.0	1	0.0625	<500	<500	6033	<0.005	
6105.50-0.05-3	(1)	985	3-9-92	3-10-92	pg. 27	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	5367				
6105.50-0.05-3	(2)	986	3-9-92	3-10-92	pg. 27	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	5318	5343	6507	0.04	82
6105.50-0.05-4	(1)	987	3-9-92	3-10-92	pg. 27	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	6347				
6105.50-0.05-4	(2)	988	3-9-92	3-10-92	pg. 27	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	6717	6532	6507	0.05	100
6105.50-0.10-3	(1)	991	3-9-92	3-10-92	pg. 27	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	11278				
6105.50-0.10-3	(2)	992	3-9-92	3-10-92	pg. 27	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	11199	11239	6462	0.09	87
6105.50-0.10-4	(1)	993	3-9-92	3-10-92	pg. 27	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	13117				
6105.50-0.10-4	(2)	994	3-9-92	3-10-92	pg. 27	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	12954	13036	6462	0.10	101
6105.50-0.50-3	(1)	997	3-9-92	3-10-92	pg. 27	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	11751				
6105.50-0.50-3	(2)	998	3-9-92	3-10-92	pg. 27	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	12054	11903	6707	0.44	89
6105.50-0.50-4	(1)	999	3-9-92	3-10-92	pg. 27	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	14686				
6105.50-0.50-4	(2)	1000	3-9-92	3-10-92	pg. 27	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	13459	14073	6707	0.52	105

CALCULATIONS PERFORMED BY: Violeta Burgos

CALCULATIONS CHECKED BY: Tarun Mehta

TABLE 2A: DETAILED RECOVERY DATA FOR METHOD VALIDATION OF CL 92,553
IN CANOLA GREEN FORAGE

COMMODITY: CANOLA GREEN FORAGE
PRODUCT: CL 92,553
ANALYTE: CL 92,553

METHOD USED: M1930.01
RESPONSE MEASURED AS: INTEGRATOR UNITS (HEIGHT)*
CHEMALYSIS PROJECT NO.: 910113

$$PPM = \frac{AVG\ R(SAMP) \times (V1) \times (V3) \times (V5) \times C(STD) \times DF}{AVG\ R(STD) \times (W) \times (V2) \times (V4)}$$

$$\%RECOVERY = \frac{(PPM\ FOUND\ IN\ FORTIFIED\ CONTROL - AVG\ PPM\ FOUND\ IN\ CONTROL) \times 100}{PPM\ ADDED}$$

NOTE: V4 = V5 = 1ul, thus cancel out of the ppm equation.
**Client ID Control AC 6794.81A

SAMPLE IDENTIFICATION					FORTIFICATION				SAMPLE PREPARATION					SAMPLE ANALYSIS AND RESULTS					
SAMPLE IDENTIFICATION**		RUN NUMBER	DATE EXTRACTED	DATE ANALYZED	NOTEBOOK #118	FV (ml)	FC ug/ml	PPM ADDED	W (gm)	V1 (ml)	V2 (ml)	V3 (ml)	DIL. FAC.	C(STD) (ug/ml)	* R(SAMP)	AVG R(SAMP)	* AVG R(STD)	APPARENT PPM FOUND	PERCENT RECOVERY
6794.81A-1	(1)	709	2-25-92	2-27-92	pg. 22	--	--	--	10	400	50	1.0	1	0.0625	2096				
6794.81A-1	(2)	710	2-25-92	2-27-92	pg. 22	--	--	--	10	400	50	1.0	1	0.0625	2088	2092	10183	0.010	
6794.81A-2	(1)	711	2-25-92	2-27-92	pg. 22	--	--	--	10	400	50	1.0	1	0.0625	2406				
6794.81A-2	(2)	712	2-25-92	2-27-92	pg. 22	--	--	--	10	400	50	1.0	1	0.0625	2411	2409	10183	0.012	
6794.81A-0.05-1	(1)	719	2-25-92	2-27-92	pg. 22	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	11152				
6794.81A-0.05-1	(2)	720	2-25-92	2-27-92	pg. 22	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	11968	11560	10273	0.06	113
6794.81A-0.05-2	(1)	721	2-25-92	2-27-92	pg. 22	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	11072				
6794.81A-0.05-2	(2)	722	2-25-92	2-27-92	pg. 22	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	11077	11075	10273	0.05	108
6794.81A-0.10-1	(1)	725	2-25-92	2-27-92	pg. 22	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	21166				
6794.81A-0.10-1	(2)	726	2-25-92	2-27-92	pg. 22	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	21086	21126	10287	0.10	103
6794.81A-0.10-2	(1)	727	2-25-92	2-27-92	pg. 22	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	16941				
6794.81A-0.10-2	(2)	728	2-25-92	2-27-92	pg. 22	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	17335	17138	10287	0.08	83
6794.81A-0.50-1	(1)	731	2-25-92	2-27-92	pg. 22	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	17170				
6794.81A-0.50-1	(2)	732	2-25-92	2-27-92	pg. 22	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	16991	17081	10380	0.41	82
6794.81A-0.50-2	(1)	733	2-25-92	2-27-92	pg. 22	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	15831				
6794.81A-0.50-2	(2)	734	2-25-92	2-27-92	pg. 22	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	15676	15754	10380	0.38	76

CALCULATIONS PERFORMED BY: Violeta Burgos

CALCULATIONS CHECKED BY: Karen Mehta

TABLE 2B: DETAILED RECOVERY DATA FOR METHOD VALIDATION OF CL 202,347 IN CANOLA GREEN FORAGE (page 1 of 2)

COMMODITY: CANOLA GREEN FORAGE
PRODUCT: CL 92,553
ANALYTE: CL 202,347

METHOD USED: M1930.01
RESPONSE MEASURED AS: INTEGRATOR UNITS (HEIGHT)*
CHEMALYSIS PROJECT NO.: 910113

$$PPM = \frac{AVG\ R(SAMP) \times (V1) \times (V3) \times (V5) \times C(STD) \times DF}{AVG\ R(STD) \times (W) \times (V2) \times (V4)}$$

$$\%RECOVERY = \frac{(PPM\ FOUND\ IN\ FORTIFIED\ CONTROL - AVG\ PPM\ FOUND\ IN\ CONTROL) \times 100}{PPM\ ADDED}$$

NOTE: V4 = V5 = 1ul, thus cancel out of the ppm equation.
**Client ID Control AC 6794.81A

SAMPLE IDENTIFICATION					FORTIFICATION			SAMPLE PREPARATION					SAMPLE ANALYSIS AND RESULTS					
SAMPLE IDENTIFICATION**	RUN NUMBER	DATE EXTRACTED	DATE ANALYZED	NOTEBOOK #118	FV (ml)	FC ug/ml	PPM ADDED	W (gm)	V1 (ml)	V2 (ml)	V3 (ml)	DIL. FAC.	C(STD) (ug/ml)	* R(SAMP)	AVG R(SAMP)	* AVG R(STD)	APPARENT PPM FOUND	PERCENT RECOVERY
6794.81A-1 (1)	685	2-25-92	2-26-92	pg. 22	--	--	--	10	400	50	1.0	1	0.0625	<400				
6794.81A-1 (2)	686	2-25-92	2-26-92	pg. 22	--	--	--	10	400	50	1.0	1	0.0625	<400	<400	4650	<0.005	
6794.81A-2 (1)	687	2-25-92	2-26-92	pg. 22	--	--	--	10	400	50	1.0	1	0.0625	<400				
6794.81A-2 (2)	688	2-25-92	2-26-92	pg. 22	--	--	--	10	400	50	1.0	1	0.0625	<400	<400	4650	<0.005	
6794.81A-3 (1)	906	3-04-92	3-05-92	pg. 25	--	--	--	10	400	50	1.0	1	0.0625	<400				
6794.81A-3 (2)	907	3-04-92	3-05-92	pg. 25	--	--	--	10	400	50	1.0	1	0.0625	<400	<400	7077	<0.005	
6794.81A-0.05-1 (1)	703	2-25-92	2-27-92	pg. 22	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	4567				
6794.81A-0.05-1 (2)	704	2-25-92	2-27-92	pg. 22	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	4751	4659	3866	0.06	121
6794.81A-0.05-2 (1)	705	2-25-92	2-27-92	pg. 22	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	4227				
6794.81A-0.05-2 (2)	706	2-25-92	2-27-92	pg. 22	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	4736	4482	3866	0.06	116

CALCULATIONS PERFORMED BY: Violeta Bueno

CALCULATIONS CHECKED BY: Tarung Mehta

TABLE 2B: DETAILED RECOVERY DATA FOR METHOD VALIDATION OF CL 202,347
IN CANOLA GREEN FORAGE (page 2 of 2)

COMMODITY: CANOLA GREEN FORAGE
PRODUCT: CL 92,553
ANALYTE: CL 202,347

METHOD USED: M1930.01
RESPONSE MEASURED AS: INTEGRATOR UNITS (HEIGHT)*
CHEMALYSIS PROJECT NO.: 910113

$$PPM = \frac{AVG\ R(SAMP) \times (V1) \times (V3) \times (V5) \times C(STD) \times DF}{AVG\ R(STD) \times (W) \times (V2) \times (V4)}$$

$$\%RECOVERY = \frac{(PPM\ FOUND\ IN\ FORTIFIED\ CONTROL - AVG\ PPM\ FOUND\ IN\ CONTROL) \times 100}{PPM\ ADDED}$$

NOTE: V4 = V5 = 1ul, thus cancel out of the ppm equation.
**Client ID Control AC 6794.81A

SAMPLE IDENTIFICATION				FORTIFICATION				SAMPLE PREPARATION					SAMPLE ANALYSIS AND RESULTS				
SAMPLE IDENTIFICATION**	RUN NUMBER	DATE EXTRACTED	DATE ANALYZED	NOTEBOOK #118	FV (ml)	FC ug/ml	PPM ADDED	W (gm)	V1 (ml)	V2 (ml)	V3 (ml)	DIL. FAC.	C(STD) (ug/ml)	* R(SAMP)	AVG R(SAMP)	* AVG R(STD)	APPARENT PPM FOUND	PERCENT RECOVERY
6794.81A-0.10-1 (1)	754	2-25-92	2-28-92	pg. 22	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	14305				
6794.81A-0.10-1 (2)	755	2-25-92	2-28-92	pg. 22	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	14087	14196	3869	0.18	183
6794.81A-0.10-2 (1)	756	2-25-92	2-28-92	pg. 22	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	8922				
6794.81A-0.10-2 (2)	757	2-25-92	2-28-92	pg. 22	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	8594	8758	3869	0.11	113
6794.81A-0.10-3 (1)	908	3-04-92	3-05-92	pg. 25	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	12464				
6794.81A-0.10-3 (2)	909	3-04-92	3-05-92	pg. 25	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	11868	12166	7077	0.09	86
6794.81A-0.10-4 (1)	912	3-04-92	3-05-92	pg. 25	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	10033				
6794.81A-0.10-4 (2)	913	3-04-92	3-05-92	pg. 25	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	10065	10049	7114	0.07	71
6794.81A-0.50-1 (1)	748	2-25-92	2-28-92	pg. 22	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	7403				
6794.81A-0.50-1 (2)	749	2-25-92	2-28-92	pg. 22	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	7191	7297	3694	0.49	99
6794.81A-0.50-2 (1)	750	2-25-92	2-28-92	pg. 22	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	4881				
6794.81A-0.50-2 (2)	751	2-25-92	2-28-92	pg. 22	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	4894	4888	3694	0.33	66
6794.81A-0.50-3 (1)	914	3-04-92	3-05-92	pg. 25	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	13349				
6794.81A-0.50-3 (2)	915	3-04-92	3-05-92	pg. 25	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	13173	13261	7114	0.47	93
6794.81A-0.50-4 (1)	932	3-04-92	3-06-92	pg. 25	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	13109				
6794.81A-0.50-4 (2)	933	3-04-92	3-06-92	pg. 25	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	12078	12594	7169	0.44	88

@ Not used in statistical calculations.

CALCULATIONS PERFORMED BY: Violeta Bulos

CALCULATIONS CHECKED BY: Karen Krebs

TABLE 3A: DETAILED RECOVERY DATA FOR METHOD VALIDATION OF CL 92,553 IN CANOLA HAY

COMMODITY: CANOLA HAY
PRODUCT: CL 92,553
ANALYTE: CL 92,553

METHOD USED: M1930.01
RESPONSE MEASURED AS: INTEGRATOR UNITS (HEIGHT)*
CHEMALYSIS PROJECT NO.: 910113

$$PPM = \frac{AVG\ R(SAMP) \times (V1) \times (V3) \times (V5) \times C(STD) \times DF}{AVG\ R(STD) \times (W) \times (V2) \times (V4)}$$

$$\%RECOVERY = \frac{(PPM\ FOUND\ IN\ FORTIFIED\ CONTROL - AVG\ PPM\ FOUND\ IN\ CONTROL) \times 100}{PPM\ ADDED}$$

NOTE: V4 = V5 = 1ul, thus cancel out of the ppm equation.
**Client ID Control AC 6794.81B

SAMPLE IDENTIFICATION					FORTIFICATION				SAMPLE PREPARATION					SAMPLE ANALYSIS AND RESULTS					
SAMPLE IDENTIFICATION**		RUN NUMBER	DATE EXTRACTED	DATE ANALYZED	NOTEBOOK #118	FV (ml)	FC ug/ml	PPM ADDED	W (gm)	V1 (ml)	V2 (ml)	V3 (ml)	DIL. FAC.	C(STD) (ug/ml)	* R(SAMP)	AVG R(SAMP)	* AVG R(STD)	APPARENT PPM FOUND	PERCENT RECOVERY
6794.81B-1	(1)	862	3-2-92	3-4-92	pg. 24	--	--	--	10	400	50	1.0	1	0.0625	1445				
6794.81B-1	(2)	863	3-2-92	3-4-92	pg. 24	--	--	--	10	400	50	1.0	1	0.0625	1448	1447	9132	0.008	
6794.81B-2	(1)	864	3-2-92	3-4-92	pg. 24	--	--	--	10	400	50	1.0	1	0.0625	1730				
6794.81B-2	(2)	865	3-2-92	3-4-92	pg. 24	--	--	--	10	400	50	1.0	1	0.0625	1750	1740	9132	0.010	
6794.81B-0.05-1	(1)	868	3-2-92	3-4-92	pg. 24	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	9708				
6794.81B-0.05-1	(2)	869	3-2-92	3-4-92	pg. 24	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	9539	9624	9283	0.05	104
6794.81B-0.05-2	(1)	870	3-2-92	3-4-92	pg. 24	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	8273				
6794.81B-0.05-2	(2)	871	3-2-92	3-4-92	pg. 24	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	8305	8289	9283	0.04	89
6794.81B-0.10-1	(1)	874	3-2-92	3-4-92	pg. 24	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	14974				
6794.81B-0.10-1	(2)	875	3-2-92	3-4-92	pg. 24	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	14868	14921	9353	0.08	80
6794.81B-0.10-2	(1)	876	3-2-92	3-4-92	pg. 24	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	16505				
6794.81B-0.10-2	(2)	877	3-2-92	3-4-92	pg. 24	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	17049	16777	9353	0.09	90
6794.81B-0.50-1	(1)	880	3-2-92	3-4-92	pg. 24	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	13289				
6794.81B-0.50-1	(2)	881	3-2-92	3-4-92	pg. 24	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	13135	13212	9260	0.36	71
6794.81B-0.50-2	(1)	882	3-2-92	3-4-92	pg. 24	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	15492				
6794.81B-0.50-2	(2)	883	3-2-92	3-4-92	pg. 24	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	15991	15742	9260	0.42	85

CALCULATIONS PERFORMED BY: Violeta Buyo

CALCULATIONS CHECKED BY: Tasmeeta

TABLE 3B: DETAILED RECOVERY DATA FOR METHOD VALIDATION OF CL 202,347
IN CANOLA HAY (page 1 of 2)

COMMODITY: CANOLA HAY
PRODUCT: CL 92,553
ANALYTE: CL 202,347

METHOD USED: M1930.01
RESPONSE MEASURED AS: INTEGRATOR UNITS (HEIGHT)*
CHEMALYSIS PROJECT NO.: 910113

$$PPM = \frac{AVG\ R(SAMP) \times (V1) \times (V3) \times (V5) \times C(STD) \times DF}{AVG\ R(STD) \times (W) \times (V2) \times (V4)}$$

$$\%RECOVERY = \frac{(PPM\ FOUND\ IN\ FORTIFIED\ CONTROL - AVG\ PPM\ FOUND\ IN\ CONTROL) \times 100}{PPM\ ADDED}$$

NOTE: V4 = V5 = 1ul, thus cancel out of the ppm equation.
**Client ID Control AC 6794.81B

SAMPLE IDENTIFICATION					FORTIFICATION				SAMPLE PREPARATION					SAMPLE ANALYSIS AND RESULTS					
SAMPLE IDENTIFICATION**		RUN NUMBER	DATE EXTRACTED	DATE ANALYZED	NOTEBOOK #118	FV (ml)	FC ug/ml	PPM ADDED	W (gm)	V1 (ml)	V2 (ml)	V3 (ml)	DIL. FAC.	C(STD) (ug/ml)	* R(SAMP)	AVG R(SAMP)	* AVG R(STD)	APPARENT PPM FOUND	PERCENT RECOVERY
6794.81B-1	(1)	838	3-2-92	3-3-92	pg. 24	--	--	--	10	400	50	1.0	1	0.0625	<500				
6794.81B-1	(2)	839	3-2-92	3-3-92	pg. 24	--	--	--	10	400	50	1.0	1	0.0625	<500	<500	6780	<0.005	
6794.81B-2	(1)	840	3-2-92	3-3-92	pg. 24	--	--	--	10	400	50	1.0	1	0.0625	<500				
6794.81B-2	(2)	841	3-2-92	3-3-92	pg. 24	--	--	--	10	400	50	1.0	1	0.0625	<500	<500	6780	<0.005	
6794.81B-3	(1)	938	3-5-92	3-6-92	pg. 26	--	--	--	10	400	50	1.0	1	0.0625	<500				
6794.81B-3	(2)	939	3-5-92	3-6-92	pg. 26	--	--	--	10	400	50	1.0	1	0.0625	<500	<500	7001	<0.005	
6794.81B-0.05-1	(1)	844	3-2-92	3-3-92	pg. 24	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	4300				
6794.81B-0.05-1	(2)	845	3-2-92	3-3-92	pg. 24	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	4229	4265	6321	0.03	67
6794.81B-0.05-2	(1)	846	3-2-92	3-3-92	pg. 24	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	4600				
6794.81B-0.05-2	(2)	847	3-2-92	3-3-92	pg. 24	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	5162	4881	6321	0.04	77
6794.81B-0.05-3	(1)	964	3-5-92	3-8-92	pg. 26	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	4501				
6794.81B-0.05-3	(2)	965	3-5-92	3-8-92	pg. 26	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	4860	4681	6370	0.04	73
6794.81B-0.05-4	(1)	944	3-5-92	3-6-92	pg. 26	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	5115				
6794.81B-0.05-4	(2)	945	3-5-92	3-6-92	pg. 26	0.50	1.0	0.05	10	400	50	1.0	1	0.0625	5619	5367	6679	0.04	80

CALCULATIONS PERFORMED BY: Violeta Buyo

CALCULATIONS CHECKED BY: Tasem Mehta

TABLE 38: DETAILED RECOVERY DATA FOR METHOD VALIDATION OF CL 202,347
IN CANOLA HAY (page 2 of 2)

COMMODITY: CANOLA HAY
PRODUCT: CL 92,553
ANALYTE: CL 202,347

METHOD USED: M1930.01
RESPONSE MEASURED AS: INTEGRATOR UNITS (HEIGHT)*
CHEMALYSIS PROJECT NO.: 910113

$$PPM = \frac{AVG\ R(SAMP) \times (V1) \times (V3) \times (V5) \times C(STD) \times DF}{AVG\ R(STD) \times (W) \times (V2) \times (V4)}$$

$$\%RECOVERY = \frac{(PPM\ FOUND\ IN\ FORTIFIED\ CONTROL - AVG\ PPM\ FOUND\ IN\ CONTROL) \times 100}{PPM\ ADDED}$$

NOTE: V4 = V5 = 1ul, thus cancel out of the ppm equation.
**Client ID Control AC 6794.818

SAMPLE IDENTIFICATION				FORTIFICATION				SAMPLE PREPARATION					SAMPLE ANALYSIS AND RESULTS					
SAMPLE IDENTIFICATION**	RUN NUMBER	DATE EXTRACTED	DATE ANALYZED	NOTEBOOK #118	FV (ml)	FC ug/ml	PPM ADDED	W (gm)	V1 (ml)	V2 (ml)	V3 (ml)	DIL. FAC.	C(STD) (ug/ml)	* R(SAMP)	AVG R(SAMP)	* AVG R(STD)	APPARENT PPM FOUND	PERCENT RECOVERY
6794.818-0.10-1 (1)	850	3-2-92	3-4-92	pg. 24	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	14090				
6794.818-0.10-1 (2)	851	3-2-92	3-4-92	pg. 24	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	14092	14091	7426	0.09	95
6794.818-0.10-2 (1)	852	3-2-92	3-4-92	pg. 24	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	33618				
6794.818-0.10-2 (2)	853	3-2-92	3-4-92	pg. 24	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	34706	34162	7426	0.23	230
6794.818-0.10-3 (1)	946	3-5-92	3-6-92	pg. 26	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	10755				
6794.818-0.10-3 (2)	947	3-5-92	3-6-92	pg. 26	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	9550	10153	6679	0.08	76
6794.818-0.10-4 (1)	950	3-5-92	3-6-92	pg. 26	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	10659				
6794.818-0.10-4 (2)	951	3-5-92	3-6-92	pg. 26	1.00	1.0	0.10	10	400	50	1.0	1	0.0625	10815	10737	6629	0.08	81
6794.818-0.50-1 (1)	856	3-2-92	3-4-92	pg. 24	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	12402				
6794.818-0.50-1 (2)	857	3-2-92	3-4-92	pg. 24	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	11832	12117	7404	0.41	82
6794.818-0.50-2 (1)	858	3-2-92	3-4-92	pg. 24	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	12435				
6794.818-0.50-2 (2)	859	3-2-92	3-4-92	pg. 24	0.50	10.0	0.50	10	400	50	5.0	1	0.0625	13237	12836	7404	0.43	87

@ Not used in statistical calculations.

CALCULATIONS PERFORMED BY: Violeta Bugos

CALCULATIONS CHECKED BY: Tasneem Abdi

TABLE 4A: RECOVERY AVERAGES FOR METHOD VALIDATION OF CANOLA COMMODITIES

SPIKING LEVEL	CANOLA COMMODITY	AVE. % REC. FOR 92,553	AVE. % REC. FOR 202,347
0.05 ppm	SEED	98	91
0.10 ppm	SEED	89	94
0.50 ppm	SEED	95	97
0.05 ppm	GREEN FORAGE	111	119
0.10 ppm	GREEN FORAGE	93	90
0.50 ppm	GREEN FORAGE	79	87
0.05 ppm	HAY	97	74
0.10 ppm	HAY	85	84
0.50 ppm	HAY	78	85

TABLE 4B: OVERALL RECOVERY AVERAGES FOR METHOD VALIDATION OF CANOLA COMMODITIES

CANOLA COMMODITY	92,553		202,347	
	Average (%)	Std. Dev. (%)	Average (%)	Std. Dev. (%)
SEED	94	4 (n=6)	94	9 (n=6)
GREEN FORAGE	94	16 (n=6)	95	19 (n=9)
HAY	87	11 (n=6)	80	8 (n=9)