STUDY TITLE

AMIZOL®: A FIELD DISSIPATION STUDY FOR TERRESTRIAL USES.

Appendix E. A High Performance Liquid Chromatographic Method for Measurement of Cyanamide Residues in Soil

Data Requirement

Subdivision N: Residue Chemistry
Guideline No. 164-1
40 CFR § 158.290

Performing Laboratory:

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Laboratory Project ID #

SLI Report #93-10-4999
SLI Study # 12983-0393-6106-360
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TITLE : A High Performance Liquid Chromatographic Method for Measurement of Cyanamide Residues in Soil

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DATE: June 17, 1993

ABSTRACT: A high performance liquid chromatographic (HPLC) method is described for the analysis of cyanamide in soil. The method employs an acetone extraction of the soil followed by centrifugation, evaporation of a portion of the sample and derivatization with dansyl chloride. The derivatized sample extracts are chromatographed using gradient elution and quantified with fluorescence detection. Recoveries of cyanamide from Oregon soil fortified in the range of 0.0100-0.100 mg/kg resulted in a mean recovery of 92.1% (S.D. 16.7) with a recovery range of 65.7%-118%. Control values were all <0.00500 mg/kg. The LOD in the Oregon soil matrix is 0.00500 mg/kg with the LOQ of 0.010 mg/kg. Recoveries of cyanamide from Washington soil fortified in the range of 0.0250-0.100 mg/kg resulted in a mean recovery of 73.0% (S.D. 10.4) with a recovery range of 62.8%-102%. Recoveries from Washington samples fortified at the 0.0100 mg/kg level resulted in artificially high recoveries due to matrix contribution. Control values indicate that the Washington matrix elevates the LOD to approximately 0.0100 mg/kg with a LOQ at 0.0250 mg/kg.

INTRODUCTION

The method for determining cyanamide residue in soil is presented. The methodology employed is novel and achieves detection limits lower than previously reported. The method was validated with soil collected from sites in Oregon and Washington (soil characterization in Tables 1 and 3). The method is flexible enough to allow for the variable matrix associated with soil.

PRINCIPLE AND APPLICATION

Cyanamide is extracted and concentrated from soil by acetone partition; the soil–solvent mixture is shaken on a shaker table and then centrifuged. Following evaporation of a portion of the extract, the residues are dissolved in an acetone solution of dansyl chloride and a pH 9.0 $Na_2CO_3/NaHCO_3$ buffer solution. The solution is then heated at 50 °C for one hour. An aliquot of this solution is analyzed by gradient elution HPLC using fluorescence detection. Quantification of cyanamide is effected by linear regression analysis of the peak heights for samples and reference standards.

ANALYTICAL METHOD

Reagents

Cyanamide, Lot No. RD-467-034, 99% active ingredient, supplied by CFPI
Acetone, Burdick and Jackson "or equivalent", HPLC grade
Acetonitrile, Burdick and Jackson "or equivalent", HPLC grade
– Dansyl chloride, Aldrich Chemical, 98% a.i.
Sodium carbonate, Fisher Scientific, AR grade
– Sodium bicarbonate monohydrate, Aldrich Chemical, 99.7% a.i.
– Sodium phosphate monobasic, Mallinckrodt, 99.3% a.i.
– Sodium phosphate dibasic, Mallinckrodt, AR grade
– Dichlorodimethylsilane, EM Science
E-Pure water ®, Barnstead "or equivalent", ASTM type II

Equipment

Balance, Ohaus Galaxy 160 "or equivalent", four-place analytical balance
Flasks, volumetric, assorted sizes
Shaker table, LabLine "or equivalent"
Centrifuge, Beckman GPR (or equivalent), 0-5000 rpm
Centrifuge bottles, 200 mL, Pyrex
Heating block, Thermolyne 17600 dry bath
Graduated centrifuge tubes, Pyrex, 15 mL capacity
Pipets, volumetric, assorted sizes
Serum bottles, Wheaton, assorted sizes, with Teflon-lined lids and metal crimp caps

Syringes, Hamilton, assorted sizes

Apparatus

High Performance Liquid Chromatographic System

Liquid chromatograph solvent pumps (2), Waters Model 6000A, Waters Model 590, in series
Solvent programmer, Waters Model 680
Autosampler, Waters Model 710B Intelligent Sample Processor
Detector, Waters Model 470 programmable fluorescence detector
Integrator, Hewlett-Packard Model 3396 A
Chromatographic Column, Metachem Inertsil ODS-2, 5μm, 250 mm x 4.6 mm I.D.

High Performance Liquid Chromatographic Conditions

Mobile Phase A: 10 mM Na_2HPO_4 / 10 mM NaH_2PO_4 (aqueous)
Mobile Phase B: 55% CH_3CN : 45% 10 mM Na_2HPO_4 / 10 mM NaH_2PO_4
Gradient program:

Time (min)	Flow (ml/min)	%A	%B	Curve
----	1.20	70	30	-----
7.00	1.20	40	60	6
12.0	1.50	0	100	1
13.0	1.20	70	30	1

Injection volume: 50 μL
Excitation wavelength: 360 nm
Emission wavelength: 495 nm
Sensitivity: 1 Volt output
Filter: 0.5 second
Autosampler runtime: 20 minutes
Attenuation (integrator): 2^4
Threshold (integrator): 4
Peak width (integrator): 0.10
Attenuation (detector): 32
Gain (detector): 10

Detailed Procedure

I. Preparation of Stock, Buffer and Standard Solutions

A. Cyanamide Stock (Solutions):

1. Weigh 100 milligrams (a.i.) of cyanamide on an analytical balance.

2. Transfer the cyanamide to a 100-mL volumetric flask and dissolve to the mark with acetone.
3. In order to prepare appropriate stocks for fortification, make serial dilutions (in acetone) as follows:

Concentration of stock (mg/L)	Volume of stock used (mL)	Final volume of dilution (mL)	Final concentration of dilution (mg/L)
1000	10.0	100	100
100	10.0	100	10.0
10.0	10.0	100	1.00
1.00	10.0	100	0.100
0.100	10.0	100	0.0100

4. Transfer each stock solution to its properly labeled 100-mL amber serum vial and seal with a Teflon-lined crimp cap.
5. Store all stock solutions in a refrigerator maintained at 4°C.

B. Sodium carbonate/sodium bicarbonate buffer:

1. Prepare 0.2 M sodium carbonate by weighing 2.12 grams of anhydrous sodium carbonate (Na_2CO_3) into a 100 mL volumetric flask and dissolving with E-Pure • water.
2. Prepare 0.2M sodium bicarbonate monohydrate by weighing 1.68 grams of sodium bicarbonate ($NaH_2CO_2H_2O$) into a 100 mL volumetric flask and dissolving with E-Pure • water.
3. Prepare the carbonate/bicarbonate buffer by volumetrically combining 4.0 mL of 0.2M sodium carbonate with 46 mL of 0.2M sodium bicarbonate and diluting the solution to 200 mL with E-Pure • water.
4. Store sodium carbonate and sodium bicarbonate stocks and prepared buffer at 4°C.

C. Cyanamide Standards:

1. In order to prepare representative standards fortify 15 mL *silanized* centrifuge tubes (as specified below). To each centrifuge tube is then volumetrically added 1.0 mL of a 1.0 mg/mL acetone solution of dansyl chloride and 1.0 mL of a pH 9.0 Na_2HCO_3 / NaH_2CO_3 aqueous buffer solution. Each tube is capped, vortexed for approximately 30 seconds and then heated in a dry heating block at 50°C for one hour. The solutions are removed from the bath, allowed to cool to room temperature and volumetrically diluted to 10.0 mL with 50% acetone : 50% pH 9 sodium carbonate/sodium bicarbonate buffer. Fortify standards as follows:

Concentration of stock (mg/L)	Volume of stock used (µL)	Final volume of dilution (mL)	Final concentration of dilution (µg/L)
1.00	500	10.0	50.0
1.00	350	10.0	35.0
1.00	250	10.0	25.0
1.00	100	10.0	10.0
1.00	50	10.0	5.00

2. Prepare fresh standards daily.

II. Control Sample Fortification

A. Processing and Dry-Weight Determination of Soil Samples:

1. Ensure that all glassware is fully silanized with dichlorodimethylsilane prior to sample processing.
2. Rinse all glassware with reagent grade acetone.
3. Remove the appropriate sets of soil cores from the freezer. Document (in the cyanamide logbook) the time of and person responsible for the removal of cores from the freezer.
4. Determine the upper end of each soil core.
5. Using a PVC tube cutter, measure, mark and remove the top 15 cm of each core and combine the appropriate cores in a mixing bowl. Allow the soil sections to thaw.
6. Homogenize until a uniform mixture is obtained (ca 10-20 minutes).
7. Remove a 50-gram portion of the mixed soil into a tared, prelabeled, 200 mL Pyrex centrifuge bottle. Document this analytical weight.
8. Remove a second soil sample (approximately 10-15 grams) and determine its soil (dry-weight) content.

B. Quality Control Sample Fortification

1. For preparation of quality control or method validation samples, fortify each preweighed soil sample (contained in a centrifuge bottle) with cyanamide by volumetric addition of the prepared stock solutions.

III. Extraction and Derivatization

1. To each soil sample add 100 mL of acetone. Secure the centrifuge bottles to a rotary shaker table. Set the table at 250 rpm and allow samples to shake for approximately 30 minutes.
2. Transfer the bottles to a centrifuge and centrifuge at 1500 rpm for 10 minutes.
3. Volumetrically remove 2.0 mL of the acetone extract from each sample, transfer to a 15 mL centrifuge tube and evaporate to dryness (at room temperature) under a gentle stream of nitrogen.
4. To each sample, volumetrically add 1.0 mL of a 1.0 mg/mL solution of dansyl chloride (in acetone) and 1.0 mL of a pH 9.0 Na_2HCO_3 / NaH_2CO_3 aqueous buffer solution. Vortex the sample briefly in order to ensure complete mixing.
5. Place the samples in a preheated dry block and heat at 50 °C for one hour.
6. Remove the samples from the block and allow to cool to room temperature.
7. Proceed to Section IV, High Performance Liquid Chromatography.

IV. High Performance Liquid Chromatography

A. Method: High performance liquid chromatographic conditions for the analysis of derivatized cyanamide standards and samples have been determined. Close adherence to these parameters is necessary in order to obtain adequate sensitivity and resolution.

B. Analysis:

1. Prepare standard solutions containing dansyl cyanamide. Standard solution concentrations used for the recovery study were 5.00, 0.0100, 25.0, 35.0 and 50.0 µg/L.
2. Inject 50µL of the 5.00 µg/L standard solution. Identify the dansyl cyanamide peak by its retention time and document the peak height. Adjust the attenuation so that the peak signal results in at least a ten percent deflection from the baseline.
3. Inject 50 µL of each of the standards, document the peak heights, and determine the coefficient of determination for the line. The coefficient of determination should be greater than or equal to 0.985.
4. Inject 50 µL of several samples. In general, a full standard set should be analyzed for every 10-12 samples.
5. Inject a full complement of standards and document peak heights.
6. Repeat steps 3-5 until all samples have been injected.
7. Construct a standard curve for dansyl cyanamide by plotting peak height observed versus the concentration (µg/L) of the standard injected.
8. The standard linear regression analysis for dansyl cyanamide is used to determine the concentration in each sample.
9. In order to determine the analytical result for each sample, the following equation is

used:

Analytical Result (µg/g) = A X D.F.

where:

Analytical Result = concentration of cyanamide (µg/g)

A = concentration (µg/L) of extract from the regression analysis

D.F. = dilution factor, ratio of the final extract volume (L) to the initial sample mass (g). **NOTE:** If the initial sample mass represents a wet weight, the analytical result is expressed as µg/kg wet weight. To convert the analytical result to a dry weight basis, multiply by the wet-to-dry weight ratio determined in step II.A.8.

RESULTS AND DISCUSSION

Soil samples were fortified with cyanamide. Each sample was extracted with acetone, shaken, centrifuged, evaporated to dryness and derivatized with dansyl chloride. Cyanamide residues were analyzed by HPLC using fluorescence detection.

The method validation/recovery study was run on soil collected from Oregon and Washington and in duplicate on separate days in order to evaluate the ruggedness and relative precision of the adopted method. Linear regression analyses for the response of cyanamide standards (as the dansyl derivative) are shown in Figure 31. Accuracy and precision of the recovery from Oregon and Washington soil is presented in Figure 32.

Recoveries from Oregon soil fortified with cyanamide at 0.100 mg/kg (wet weight) ranged from 65.7% to 107% with an average recovery of 81.6% (S.D. 15.6); recoveries at 0.0500 mg/kg ranged from 68.7% to 105% with an average recovery of 88.1% (S.D. 17.8); recoveries at 0.0250 mg/kg ranged from 73.5% to 114% with an average recovery of 95.5% (S.D. 14.2); recoveries at 0.0100 mg/kg ranged from 77.2% to 118% with an average recovery of 103% (S.D. 14.3). The LOD for cyanamide was 0.00500 mg/kg, calculated as the predicted analytical resulted at one half of the LOQ (0.0100 mg/kg). The LOQ was determined as a function of the wet weight of the sample. A summary of recovery data obtained from Oregon soil is presented in Table 32. Representative chromatograms of Oregon soil extracts containing cyanamide at the 0.100, 0.0500, 0.0250, 0.0100 mg/kg and control levels are shown in Figures 33-37, respectively.

Recoveries from Washington soil fortified with cyanamide at 0.100 mg/kg (wet weight) ranged from 62.8% to 72.4% with an average recovery of 65.6 (S.D. 3.63); recoveries at 0.0500 mg/kg ranged from 71.7% to 77.3% with an average recovery of 74.3% (S.D. 2.25); recoveries at 0.0250 mg/kg ranged from 63.0% to 102% with an average recovery of 79.3% (S.D. 15.2); recoveries at 0.0100 mg/kg ranged from 123% to 154%

with an average recovery of 137% (S.D. 14.9). As observed in the control samples, a significant matrix interference resulted in artificially elevated recoveries at the 0.0100 mg/kg level. The LOD for cyanamide was compromised due to the matrix contribution of the Washington soil. The LOD for this soil should be considered to approximate 0.0100 mg/kg; the LOQ should be 0.0250 mg/kg. The LOQ was determined as a function of the wet weight of the sample. A summary of recovery data obtained from Washington soil is presented in Table 33. Representative chromatograms of Washington soil extracts containing cyanamide at the 0.100, 0.0500, 0.0250, 0.0100 mg/kg and control levels are shown in Figures 38-42, respectively.

SUMMARY

A procedure for the measurement of cyanamide in soil has been developed. The analytical procedure entails the extraction of cyanamide from soil, its derivatization, separation from artifacts by HPLC using gradient elution and its quantification by fluorescence detection.

Table 32. Analytical results for the recovery of cyanamide from Oregon soil.

Fortified Level (mg/kg)	Sample Mass (grams)	Level Found (mg/kg)	Percent Recovery (%)	Mean Percent Recovery (%)
0.100	50	0.09188	91.9	
0.100	50	0.1071	107	
0.100	50	0.08121	81.2	
0.100	50	0.06575	65.7	
0.100	50	0.07547	75.5	
0.100	50	0.06838	68.4	81.6
0.0500	50	0.05167	103	
0.0500	50	0.5259	105	
0.0500	50	0.05205	104	
0.0500	50	0.03582	71.6	
0.0500	50	0.03436	68.7	
0.0500	50	0.03779	75.6	88.1
0.0250	50	0.02599	104	
0.0250	50	0.02541	102	
0.0250	50	0.02850	114	
0.0250	50	0.02178	87.1	
0.0250	50	0.02322	92.9	
0.0250	50	0.01839	73.5	95.5
0.0100	50	0.01176	118	
0.0100	50	0.01092	109	
0.0100	50	0.01054	105	
0.0100	50	0.01118	112	
0.0100	50	0.009876	98.7	
0.0100	50	0.007716	77.2	103
Control	50	<0.00500	NA	
Control	50	<0.00500	NA	
Control	50	<0.00500	NA	
Control	50	<0.00500	NA	
Control	50	<0.00500	NA	
Control	50	<0.00500	NA	NA

Average Recovery (0.0100-0.100 mg/kg): 92.1%; S.D. 16.7 (CV = 18.1; N=24)
Theoretical limit of detection is 0.00500 mg/kg for a 50 gram sample.
LOQ = 0.010 mg/kg.

Table 33. Analytical results for the recovery of cyanamide from Washington soil.

Fortified Level (mg/kg)	Sample Mass (grams)	Level Found (mg/kg)	Percent Recovery (%)	Mean Percent Recovery (%)
0.100	50	0.06551	65.5	
0.100	50	0.06277	62.8	
0.100	50	0.07237	72.4	
0.100	50	0.06335	63.4	
0.100	50	0.06616	66.2	
0.100	50	0.06304	63.0	65.6
0.0500	50	0.03656	73.1	
0.0500	50	0.03586	71.7	
0.0500	50	0.03863	77.3	
0.0500	50	0.03629	72.6	
0.0500	50	0.03832	76.6	
0.0500	50	0.03718	74.4	74.3
0.0250	50	0.02314	92.6	
0.0250	50	0.01839	73.6	
0.0250	50	0.01919	76.8	
0.0250	50	0.02560	102	
0.0250	50	0.01681	67.3	
0.0250	50	0.01575	63.0	79.3
0.0100	50	0.01230	123	
0.0100	50	0.01234	123	
0.0100	50	0.01243	124	
0.0100	50	0.01539	154	
0.0100	50	0.01442	144	
0.0100	50	0.01523	152	137
Control	50	0 005187	NA	
Control	50	0.007514	NA	
Control	50	<0.00500	NA	
Control	50	<0.00500	NA	
Control	50	<0.00500	NA	
Control	50	0.005523	NA	NA

Average Recovery (0.0100-0.100 mg/kg) 89.0%; S.D. 30.4 (CV = 34.2; N = 24)
Average Recovery (0.0250-0.100 mg/kg) 73.0%; S.D. 10.4 (CV = 14.2; N = 18)
Theoretical minimum detectable concentration is 0.0100 mg/kg for a 50 gram sample.
LOQ = 0.0250 mg/kg.

Figure 31. The linear regression analysis for cyanamide standards used in the recovery study.

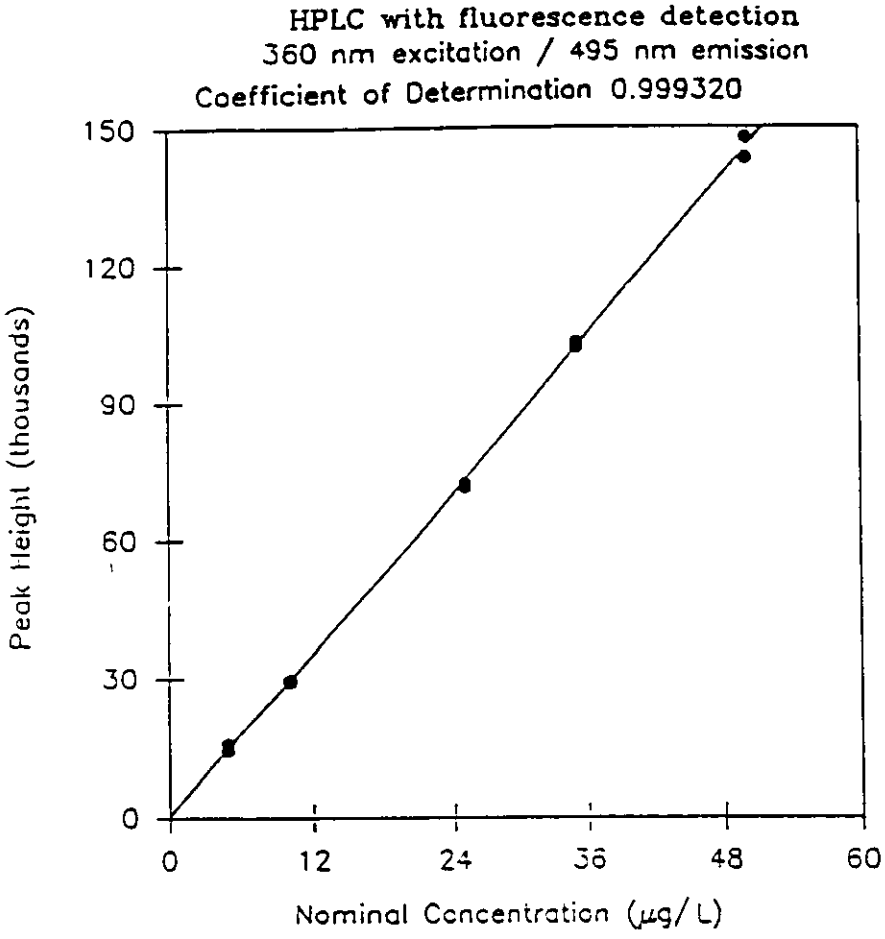


Figure 32. Mean and standard deviation representation of recovery samples of cyanamide from Oregon and Washington soil.

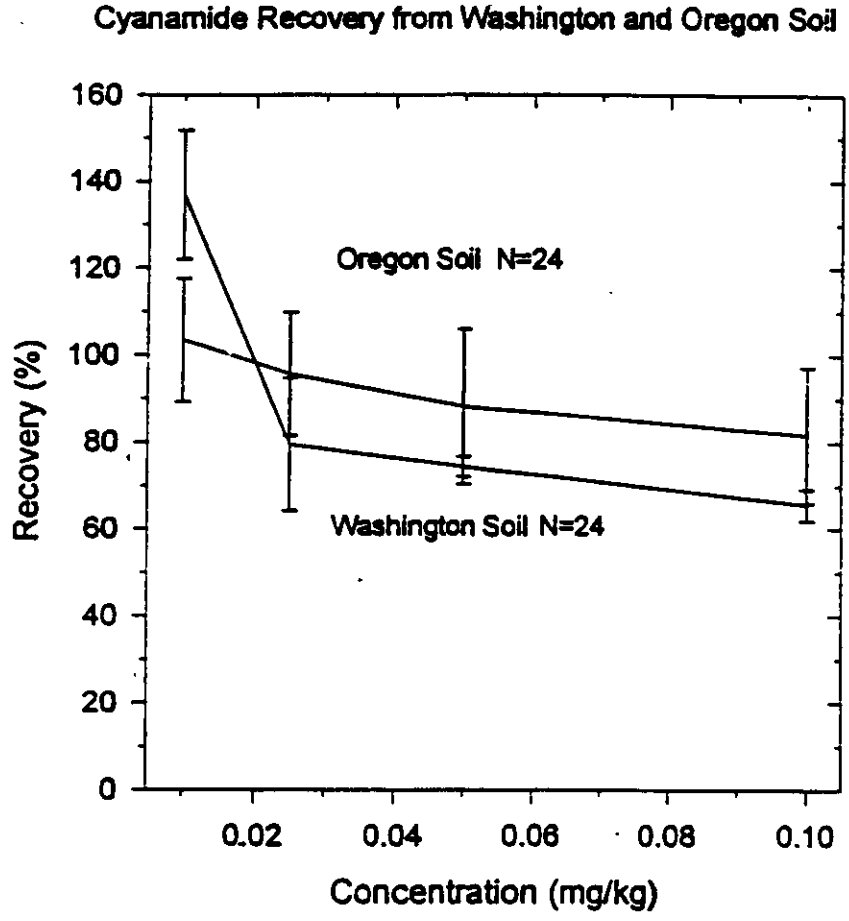
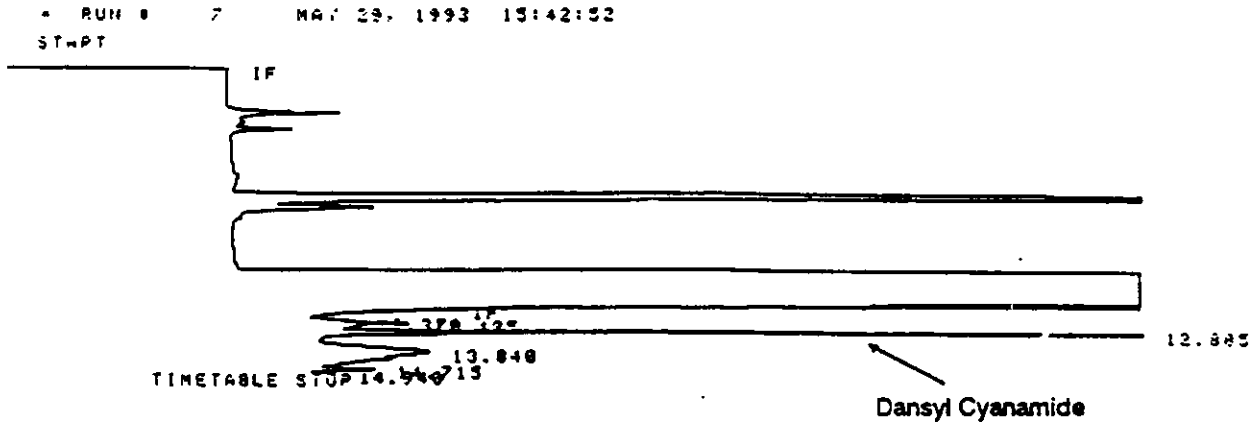


Figure 33. A representative chromatogram of an Oregon soil extract fortified as 0.100 mg/kg of cyanamide.



RUN# 7 MAY 29, 1993 15:42:52

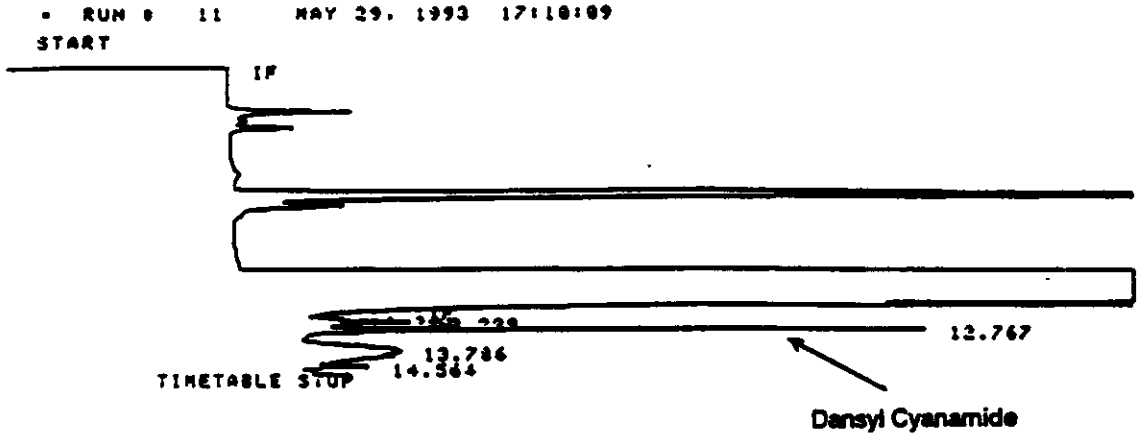
DANS-CYANAMIDE

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
12.378	PV	35618	.114	5196		.000	
12.495	VV	33309	.116	11974		.000	
12.885	VB	952552	100	159496	1R	46.221	
13.840	BV	37213-	45	13946		.000	
14.715	VP	55672	121	7681		.000	
14.949	I PH	12093	.084	2399		.000	

TOTAL AREA=1511371
MUL FACTOR=1.0000E+00

Figure 34. A representative chromatogram of an Oregon soil extract fortified as 0.0500 mg/kg of cyanamide.



RUN# 11 MAY 29, 1993 17:10:09

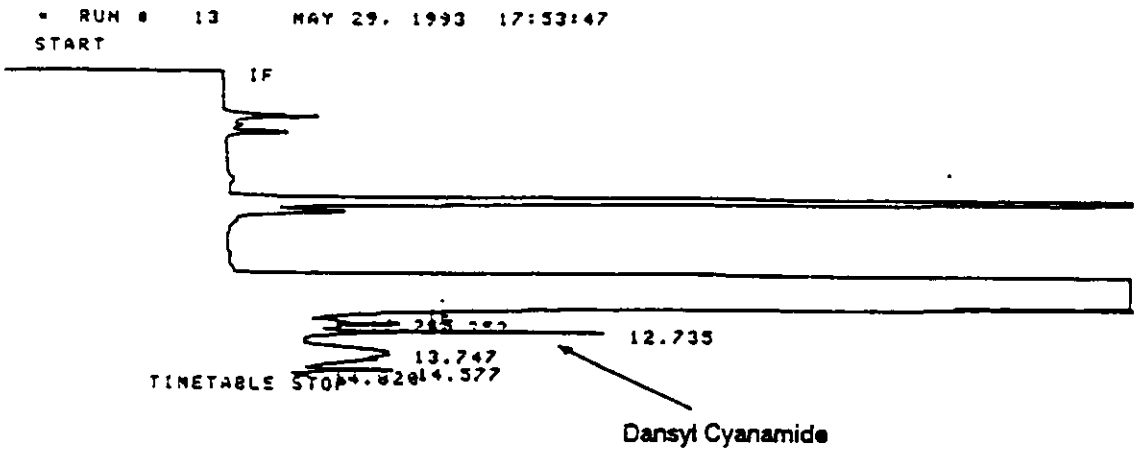
DANS-CYANAMIDE

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
12.294	PV	27315	.121	3760		.000	
12.389	VV	62550	.094	11097		.000	
12.767	BB	427875	.092	77398	IR	28.762	
13.786	BV	313314	.434	12025		.000	
14.564	VV	61983	.127	8137		.000	

TOTAL AREA= 893037
MUL FACTOR=1.0000E+00

Figure 35. A representative chromatogram of an Oregon soil extract fortified as 0.0250 mg/kg of cyanamide.



RUN# 10 MAY 29, 1993 17:53:47

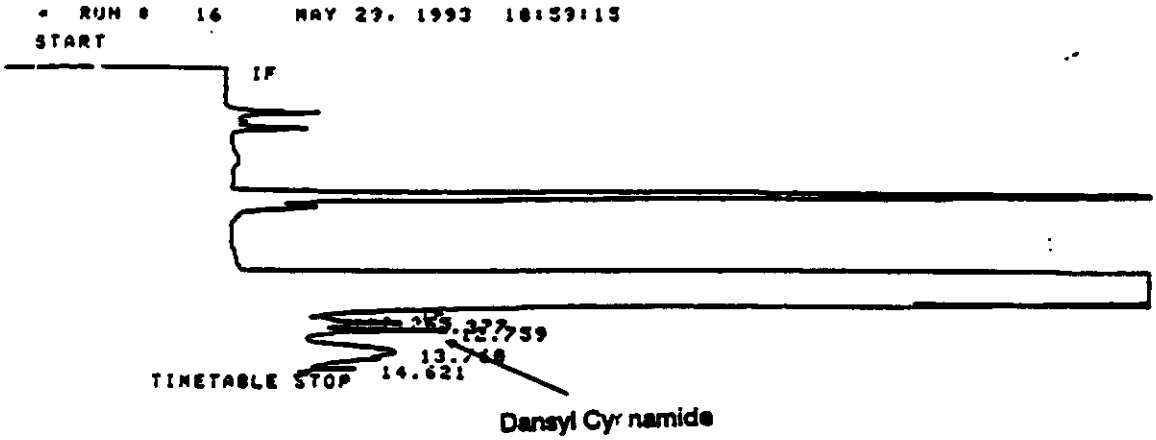
DANS-CYANAMIDE

ESTD-AREA

PT	TYPE	AREA	WIDTH	HEIGHT	C.L.#	AMOUNT	NAME
12.245	BV	10319	.112	4593		.000	
12.352	VV	79001	.103	11325		.000	
12.705	VB	340039	.106	37653		.000	
13.747	VV	327773	.481	11396	IR	15.905	
14.577	VP	96714	.103	13603		.000	
14.920	I PP	14564	.110	2213		.000	

TOTAL AREA= 773971
MUL FACTOR=1.0000E+00

Figure 36. A representative chromatogram of an Oregon soil extract fortified as 0.0100 mg/kg of cyanamide.



RUN# 16 MAY 29, 1993 13:59:15

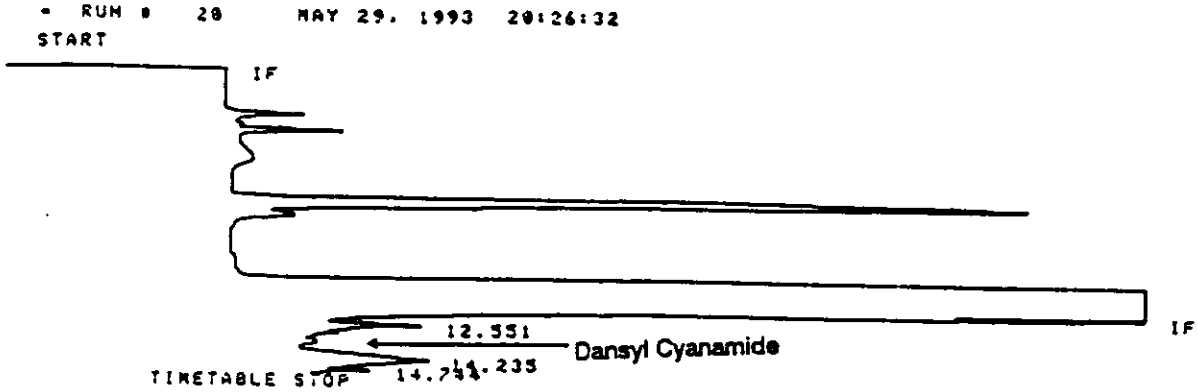
DANS-CYANAMIDE

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
12.265	BV	28805	.124	3863		.000	
12.377	VV	71625	.105	11363		.000	
12.759	VV	139485	.145	16048		.000	
13.760	VV	370073	.525	11741	1R	17.357	
14.621	VV	62530	.130	7524		.000	

TOTAL AREA= 672514
MUL FACTOR=1.0000E-00

Figure 37. A representative chromatogram of an Oregon soil extract which was not fortified with cyanamide, but concentrated to the same level as the 0.0100 mg/kg samples.



RUN# 20 MAY 29, 1993 20:26:32

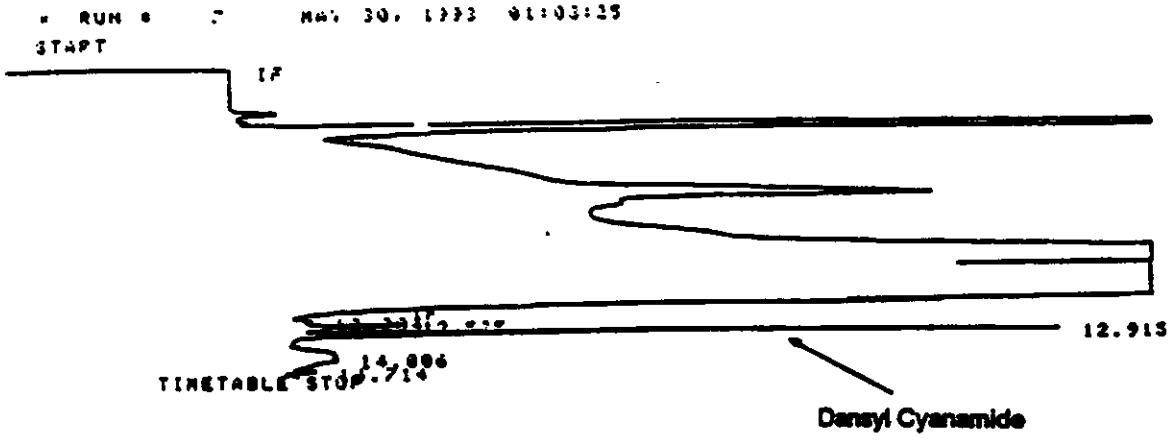
DANS-CYANAMIDE

EXTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
12.551	BV	141333	.198	11377		.000	
14.215	PV	443759	.497	15863	IF	21.923	
14.744	VP	99537	.126	7363		.000	

TOTAL AREA= 684629
MUL FACTOR=1.0000E+00

Figure 38. A representative chromatogram of a Washington soil extract fortified as 0.100 mg/kg of cyanamide.



RUN# 7 MAY 30, 1993 01:03:25

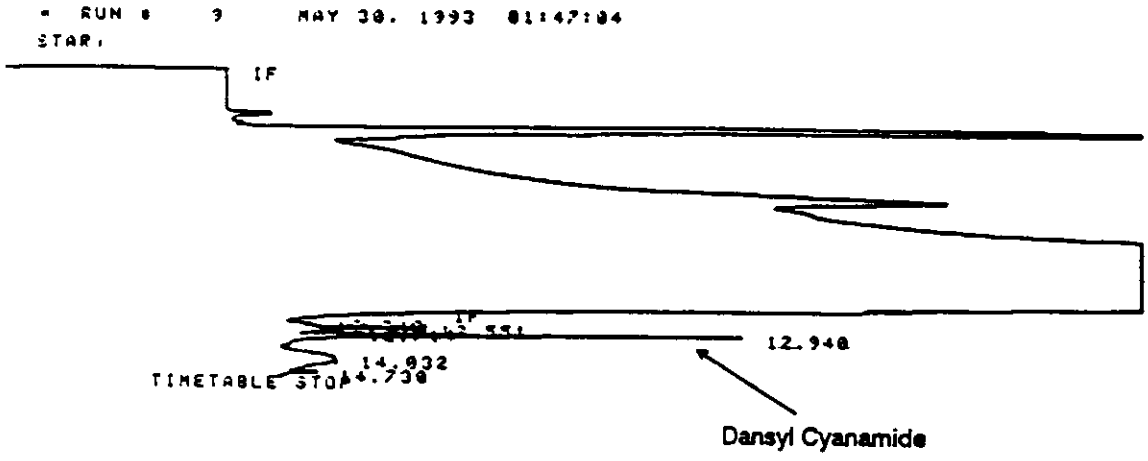
DANS-CYANAMIDE

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
12.294	BV	16371	.109	2579		.000	
12.525	VV	77342	.182	12638		.000	
12.915	VB	532439	.893	94099	1R	25.836	
14.096	PV	168342	.458	6131		.000	
14.714	VP	27358	.120	3737		.000	

TOTAL AREA= 822352
AUL FACTOR=1.0000E+00

Figure 39. A representative chromatogram of a Washington soil extract fortified as 0.0500 mg/kg of cyanamide.



RUN# 9 MAY 30, 1993 01:47:04

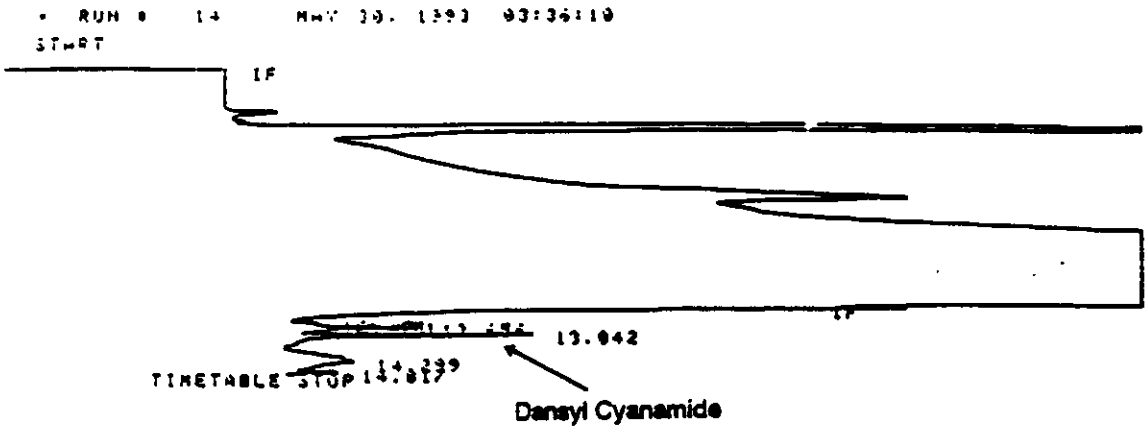
DNS-CYANAMIDE

EXTO-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CHL#	AMOUNT	NAME
12.348	PV	1[illegible]61	.102	3038		.009	
12.461	VV	15621	.066	3951		.000	
12.591	V	74439	.081	15395		.000	
12.714	VV	46263	.119	6487		.000	
12.940	VB	332950	.099	55910	1R	16.157	
14.032	PV	162782	.376	7218		.000	
14.709	VV	39541	.131	5977		.001	

TOTAL AREA= 699529
MUL FACTOR=1.0000E+00

Figure 40. A representative chromatogram of a Washington soil extract fortified as 0.0250 mg/kg of cyanamide.



RUN# 14 MAY 30, 1993 03:36:10

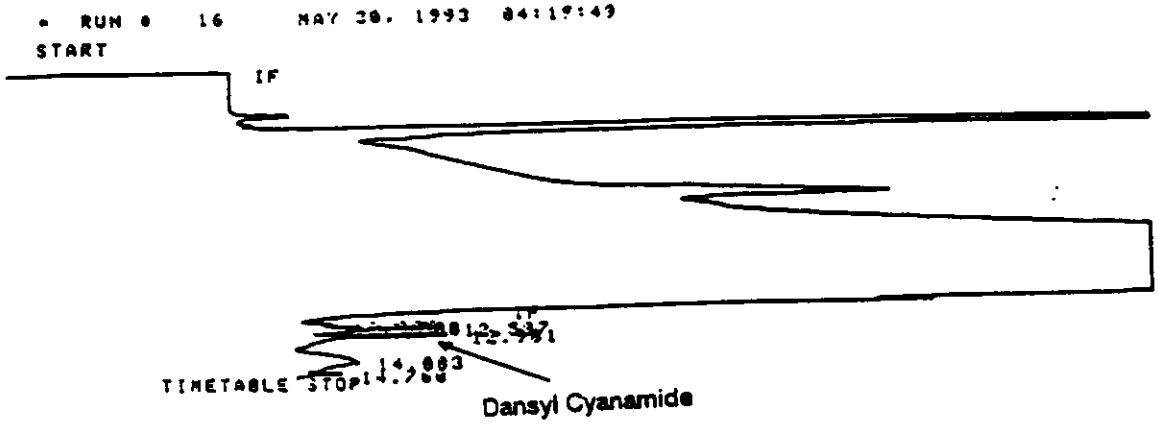
DANS-CYANAMIDE

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
12.471	PV	21214	.097	3639		.000	
12.571	VV	19942	.062	4813		.000	
12.656	VV	75538	.082	15470		.000	
13.942	VB	195338	.105	29358	IR	0.793	
14.299	PV	278498	.345	8213		.000	
14.817	I VP	40549	.113	5968		.000	

TOTAL AREA= 628179
MUL FACTOR=1.0000E+00

Figure 41. A representative chromatogram of a Washington soil extract fortified as 0.0100 mg/kg of cyanamide.



RUN# 16 MAY 30, 1993 04:19:49

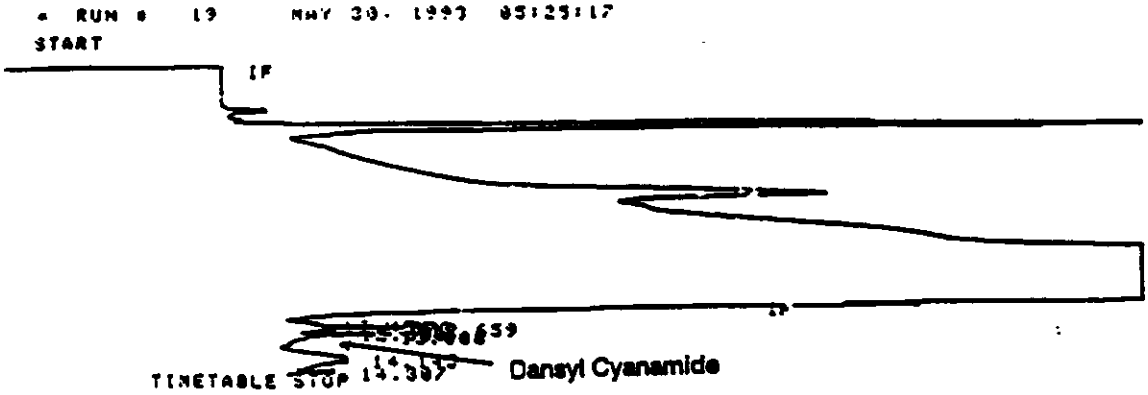
DANS-CYANAMIDE

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
12.350	BV	28334	.122	4150		.000	
12.508	VV	37992	.079	6653		.000	
12.597	VV	26737	.091	17613		.000	
12.961	VP	173996	.158	18356	IP	8.685	
14.033	PV	166837	.362	7481		.000	
14.769 I	VP	41270	.122	5721		.000	

TOTAL AREA= 536216
MUL FACTOR=1.0000E+00

Figure 42. **A representative chromatogram of a Washington soil extract which was not fortified with cyanamide, but concentrated to the same level as the 0.0100 mg/kg samples.**



RUN# 19 MAY 30, 1993 05:25:17

DANS-CYANAMIDE

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
12.454	BV	[illegible]	.129	4196		.000	
12.573	VV	28225	.069	6826		.000	
12.659	VV	75863	.084	14962		.000	
12.803	VV	52619	.135	6502		.000	
13.008	VV	105992	.154	11462		.000	
14.143	PV	158591	.331	7975	1R	7.695	
14.307	VV	45291	.120	6291		.000	

TOTAL AREA= 496811
NUL FACTOR=1.0000E-00

APPENDIX F. WEATHER DATA

Appendix F. Daily air temperature, soil temperature and precipitation collected by Scientific CR-10 Data logger at the Hillsboro, Oregon study site.

Month/ Year	Month/ Day	Air Temp. (°F)		Soil Temp. (°F)		Precipitation (inches)
		Min	Max	Min	Max	
JUNE						
1993	601	51.2	67.7	59.7	67.3	0.18
1993	602	48.3	72.2	58.5	71.8	0.05
1993	603	44.1	70.1	57.5	70.1	0.03
1993	604	52.0	73.0	60.1	71.1	0.20
1993	605	55.4	73.6	61.9	69.0	0.18
1993	606	54.8	68.6	61.2	66.3	0.00
1993	607	46.9	71.8	58.5	70.4	0.00
1993	608	48.7	69.1	58.0	70.8	0.00
1993	609	47.8	70.2	60.4	68.4	0.07
1993	610	46.1	67.3	57.7	66.1	0.00
1993	611	46.2	68.3	56.4	67.2	0.13
1993	612	47.3	71.0	56.6	72.0	0.00
1993	613	42.2	78.3	56.3	71.4	0.00
1993	614	53.9	79.3	61.0	72.4	0.10
1993	615	49.8	69.3	61.3	67.5	0.00
1993	616	48.8	77.7	59.3	72.0	0.00
1993	617	51.1	80.2	59.9	76.2	0.33
1993	618	52.7	86.5	61.1	78.3	0.00
1993	619	52.2	80.1	60.8	77.5	0.00
1993	620	46.0	82.6	59.6	78.4	0.00
1993	621	48.1	71.6	61.0	69.4	0.05
1993	622	46.2	67.4	56.7	68.8	0.50
1993	623	41.4	73.9	55.2	71.9	0.00
1993	624	44.3	82.8	57.2	77.3	0.00
1993	625	51.4	89.2	61.6	80.6	0.00
1993	626	55.5	79.5	64.2	74.2	0.01
1993	627	51.6	71.0	61.8	71.4	0.01
1993	628	46.6	70.7	59.1	68.5	0.06
1993	629	47.7	73.1	58.0	74.6	0.13
1993	630	51.6	77.2	60.6	75.5	0.00

Appendix F. Daily air temperature, soil temperature and precipitation collected by Scientific CR-10 Data logger at the Hillsboro, Oregon study site.

Month/ Year	Month/ Day	Air Temp. (°F)		Soil Temp. (°F)		Precipitation (inches)
		Min	Max	Min	Max	
JULY						
1993	701	51.8	74.0	61.8	72.3	0.00
1993	702	52.1	75.2	62.0	74.3	0.01
1993	703	51.2	77.0	60.7	71.5	0.00
1993	704	50.1	73.7	59.0	73.5	0.24
1993	705	48.3	74.0	59.7	72.5	0.00
1993	706	50.5	73.6	60.3	71.5	0.00
1993	707	42.9	77.7	56.3	75.8	0.00
1993	708	47.1	84.6	59.0	80.2	0.00
1993	709	42.5	74.5	57.7	70.4	0.00
1993	710	44.4	78.5	56.7	78.1	0.00
1993	711	47.6	72.5	61.0	70.6	0.00
1993	712	45.8	70.7	58.8	68.3	0.00
1993	713	53.1	72.3	60.1	69.9	0.23
1993	714	51.9	72.8	60.3	69.9	0.05
1993	715	53.6	74.0	60.8	69.9	0.00
1993	716	53.1	65.9	60.2	66.2	0.42
1993	717	53.8	73.2	60.4	69.0	0.05
1993	718	47.9	82.0	58.7	77.2	0.00
1993	719	54.8	70.1	62.5	69.9	0.11
1993	720	53.7	75.5	61.8	71.3	0.02
1993	721	51.9	73.5	60.8	69.5	0.05
1993	722	58.0	75.3	61.5	69.2	0.00
1993	723	53.3	73.3	62.2	70.7	0.00
1993	724	52.1	68.2	60.5	67.0	0.00
1993	725	54.9	76.6	60.4	71.7	0.00
1993	726	49.6	81.6	59.3	78.7	0.00
1993	727	49.6	88.4	61.3	82.0	0.00
1993	728	50.5	67.5	62.9	70.9	0.02
1993	729	47.4	70.9	58.4	68.6	0.02
1993	730	47.7	81.6	57.5	78.7	0.00
1993	731	49.5	83.8	60.7	79.4	0.11

Appendix F. Daily air temperature, soil temperature and precipitation collected by Scientific CR-10 Data logger at the Hillsboro, Oregon study site.

Month/ Year	Month/ Day	Air Temp. (°F)		Soil Temp. (°F)		Precipitation (inches)
		Min	Max	Min	Max	
AUGUST						
1993	801	54.2	90.2	63.1	79.7	0.00
1993	802	55.3	95.1	63.5	83.6	0.00
1993	803	56.9	98.9	65.7	86.6	0.00
1993	804	60.3	97.2	68.5	86.0	0.34
1993	805	58.4	93.5	68.4	85.3	0.36
1993	806	57.3	81.4	68.7	78.1	0.01
1993	807	58.5	76.5	65.9	75.1	0.00
1993	808	52.2	79.0	64.9	74.8	0.00
1993	809	52.7	79.3	63.2	73.0	0.00
1993	810	49.8	79.4	61.3	76.1	0.00
1993	811	48.8	79.9	61.0	77.8	0.00
1993	812	50.8	76.2	62.0	74.1	0.00
1993	813	55.8	67.4	62.9	68.1	0.00
1993	814	54.5	69.9	61.5	68.0	0.09
1993	815	54.5	75.0	61.9	72.1	0.00
1993	816	52.7	79.0	62.1	70.7	0.05
1993	817	47.7	77.6	58.5	74.0	0.26
1993	818	50.7	85.4	60.8	76.6	0.04
1993	819	61.8	85.9	66.0	77.1	0.03
1993	820	56.2	76.4	65.6	72.9	0.00
1993	821	56.6	81.3	64.2	73.9	0.00
1993	822	50.0	87.3	61.8	75.2	0.00
1993	823	46.8	77.7	63.3	72.7	0.01
1993	824	40.7	72.4	56.4	69.0	0.81
1993	825	40.4	70.8	53.8	69.0	0.27
1993	826	42.5	82.2	55.3	71.8	0.00
1993	827	46.5	80.1	57.6	72.8	0.00
1993	828	47.0	74.7	57.6	69.6	0.00
1993	829	45.5	82.3	56.7	72.9	0.00
1993	830	46.5	86.1	57.6	74.4	0.00
1993	831	45.9	93.1	57.7	75.2	0.00

Appendix F. Daily air temperature, soil temperature and precipitation collected by Scientific CR-10 Data logger at the Hillsboro, Oregon study site.

Month/ Year	Month/ Day	Air Temp. (°F)		Soil Temp. (°F)		Precipitation (inches)
		Min	Max	Min	Max	
SEPTEMBER						
1993	901	48.8	79.8	59.8	75.4	0.00
1993	902	48.3	88.0	59.9	76.0	0.00
1993	903	50.8	90.1	61.3	77.3	0.00
1993	904	49.9	80.9	62.3	77.1	0.00
1993	905	52.8	80.8	63.3	76.1	0.00
199_	906	51.3	91.8	61.4	77.9	0.44
1993	907	54.5	93.5	63.3	78.5	0.00
1993	908	53.3	93.0	63.2	78.7	0.00
1993	909	50.4	93.9	61.9	79.1	0.00
1993	910	50.0	91.8	61.9	78.7	0.00
1993	911	45.1	75.1	59.9	71.3	0.00
1993	912	47.6	75.5	58.9	73.2	0.00
1993	913	41.0	81.4	55.4	72.7	0.00
1993	914	49.5	76.5	58.3	68.8	0.00
1993	915	49.6	74.7	59.8	70.1	0.00
1993	916	42.3	79.0	54.9	71.9	0.00
1993	917	43.5	81.4	55.7	72.5	0.00
1993	918	42.3	71.1	55.2	66.5	0.00
1993	919	40.0	70.9	56.4	65.9	0.00
1993	920	39.0	67.8	53.1	62.7	0.00
1993	921	34.4	71.8	49.7	67.2	0.00
1993	922	35.9	75.9	50.1	68.4	0.00
1993	923	37.1	78.9	51.0	68.9	0.15
1993	924	39.5	76.5	52.1	65.3	0.00
1993	925	42.1	78.5	52.7	68.4	0.00
1993	926	42.3	86.2	53.0	70.4	0.00
1993	927	42.9	86.6	54.0	70.9	0.72
1993	928	44.7	85.6	56.0	70.0	0.00
1993	929	46.8	93.9	56.2	72.1	0.00
1993	930	49.6	83.7	57.9	70.8	0.00

Appendix F. Daily air temperature, soil temperature and precipitation collected by Scientific CR-10 Data logger at the Hillsboro, Oregon study site.

Month/ Year	Month/ Day	Air Temp. (°F)		Soil Temp. (°F)		Precipitation (inches)
		Min	Max	Min	Max	
OCTOBER						
1993	1001	43.0	80.1	55.5	70.8	0.02
1993	1002	44.2	89.7	55.6	71.3	0.00
1993	1003	45.0	87.1	56.5	71.5	0.00
1993	1004	44.2	83.9	55.9	69.6	0.00
1993	1005	44.8	70.7	56.3	64.3	2.49
1993	1006	49.2	62.4	56.2	62.0	0.52
1993	1007	43.8	68.6	54.8	63.8	0.01
1993	1008	39 5	70.4	53.0	61.9	0.01
1993	1009	41.6	76.0	51.0	63.5	0.00
1993	1010	40.7	71.8	51.4	61.3	0.00
1993	1011	43.1	67.9	52.5	61.0	0.01
1993	1012	50.2	68.4	55.4	61.5	0.27
1993	1013	51.8	70.3	57.4	63.2	0.00
1993	1014	51.4	60.9	56.6	60.8	0.14
1993	1015	47.2	67.2	56.4	62.2	0.29
1993	1016	43.8	62.9	53.7	59.9	0.02
1993	1017	41.9	64.1	52.5	60.2	0.00
1993	1018	36.9	62.2	50.0	58.0	0.00
1993	1019	41.7	67.9	51.9	61.4	0.00
1993	1020	36.3	73.3	48.6	60.1	0.00
1993	1021	43.4	65.9	52.4	59.3	0.00
1993	1022	40.8	65.4	50.9	58.4	0.00
1993	1023	43.0	69.6	51.8	59.8	0.04
1993	1024	37.0	66.0	49.7	58.3	0.00
1993	1025	35.6	67.6	47.6	56.9	0.00
1993	1026	40.7	71.3	48.1	56.9	0.00
1993	1027	34.6	70.6	46.8	55.7	0.00
1993	1028	41.6	73.6	49.5	58.5	0.00
1993	1029	39.7	71.2	49.3	58.1	0.00
1993	1030	35.2	73.1	47.6	56.6	0.00
1993	1031	36.7	65.5	50.6	56.9	0.27

Appendix F. Daily air temperature, soil temperature and percipitation collected by the Campbell Scientific CR-21 Data logger at the Moses Lake, Washington study site.

Month/ Year	Month/ Day	Julian Day	Air Temp. (°F)	Soil Temp. (°F)	Precipitation (inches)
JUNE					
1993	616	167	66.4	65.7	0
1993	617	168	64.08	71.3	0.04
1993	618	169	70.1	73.7	0
1993	619	170	74.1	75.6	0
1993	620	171	68.82	69.3	0.04
1993	621	172	62.6	68.4	0.28
1993	622	173	54.85	61.9	0
1993	623	174	57.17	60.7	0
1993	624	175	59.44	66.79	0
1993	625	176	65.36	72.5	0
1993	626	177	72.2	76.8	0
1993	627	178	76.4	81	0
1993	628	179	64.22	74.8	0
1993	629	180	61.09	73	0
1993	630	181	62.93	75.4	0

Appendix F. Daily air temperature, soil temperature and percipitation collected by the Campbell Scientific CR-21 Data logger at the Moses Lake, Washington study site.

Month/ Year	Month/ Day	Julian Day	Air Temp. (°F)	Soil Temp. (°F)	Precipitation (inches)
JULY					
1993	701	182	64.18	73.6	0.03
1993	702	183	65.21	73	0
1993	703	184	62.19	73.8	0
1993	704	185	67.58	75.6	0
1993	705	186	69.41	79.8	0
1993	706	187	68.73	78.8	0
1993	707	188	66.54	78.9	0.07
1993	708	189	65.02	75	0.24
1993	709	190	70.6	74.8	0
1993	710	191	70.3	77.2	0
1993	711	192	68.92	78.8	0
1993	712	193	64.6	77.8	0
1993	713	194	62.13	73.6	0
1993	714	195	66.88	77.5	0.02
1993	715	196	62.19	75	0.02
1993	716	197	64.71	76.5	0
1993	717	198	64.86	76.6	0
1993	718	199	64.21	75.6	0
1993	719	200	67.81	77.6	0
1993	720	201	68.48	76.4	0
1993	721	202	65.98	76.6	0.01
1993	722	203	67.41	78.1	0
1993	723	204	67.01	75.3	0.03
1993	724	205	68.83	77	0
1993	725	206	67.96	78.2	0
1993	726	207	64.61	72.8	0.06
1993	727	208	72.2	77.6	0.01
1993	728	209	74	79	0
1993	729	210	77.3	81.7	0
1993	730	211	67.21	78.6	0
1993	731	212	61.77	71.3	0.11

Appendix F. Daily air temperature, soil temperature and percipitation collected by the Campbell Scientific CR-21 Data logger at the Moses Lake, Washington study site.

Month/ Year	Month/ Day	Julian Day	Air Temp. (°F)	Soil Temp. (°F)	Precipitation (inches)
AUGUST					
1993	801	213	69.07	72.1	0
1993	802	214	73.4	77.2	0
1993	803	215	75.9	80	0
1993	804	216	76.8	80.6	0
1993	805	217	76.8	81.1	0
1993	806	218	78.2	82.7	0
1993	807	219	76.9	83.4	0
1993	808	220	74.2	81.9	0
1993	809	221	70.5	79.9	0
1993	810	222	69.75	79.1	0
1993	811	223	70.2	78.8	0
1993	812	224	68.84	78.6	0
1993	813	225	69.85	78.3	0
1993	814	226	69.28	78.5	0
1993	815	227	66.43	76.8	0
1993	816	228	68.12	78.4	0
1993	817	229	61.42	69.79	0.3
1993	818	230	69.16	71.6	0.01
1993	819	231	72.7	74	0
1993	820	232	75.8	76.9	0
1993	821	233	74.7	77.4	0
1993	822	234	67.96	73.7	0
1993	823	235	69.14	75	0
1993	824	236	67.61	74.7	0
1993	825	237	59.21	70.8	0
1993	826	238	57.8	69.6	0
1993	827	239	59.89	71.5	0
1993	828	240	64.14	71.4	0
1993	829	241	59.47	67.71	0.01
1993	830	242	60.82	69.39	0
1993	831	243	65.03	71.9	0

Appendix F. Daily air temperature, soil temperature and percipitation collected by the Campbell Scientific CR-21 Data logger at the Moses Lake, Washington study site.

Month/ Year	Month/ Day	Julian Day	Air Temp. (°F)	Soil Temp. (°F)	Precipitation (inches)
SEPTEMBER					
1993	901	244	64.67	71.3	0
1993	902	245	66.28	72.5	0
1993	903	246	69.02	74.7	0
1993	904	247	71.4	76.2	0
1993	905	248	71.8	77.2	0
1993	906	249	72.5	76.7	0
1993	907	250	71.8	77.1	0
1993	908	251	70.7	76.4	0
1993	909	252	71.9	77.2	0
1993	910	253	70.4	76.8	0
1993	911	254	69.2	76.1	0
1993	912	255	62.09	68.87	0
1993	913	256	57.46	66.57	0
1993	914	257	55.34	66.13	0
1993	915	258	53.17	62.12	0.04
1993	916	259	56.01	62.09	0
1993	917	260	51.43	60.62	0
1993	918	261	55.36	59.08	0
1993	919	262	55.7	58.89	0
1993	920	263	54.34	57.75	0
1993	921	264	46.74	52.88	0.09
1993	922	265	49.47	53.9	0
1993	923	266	49.78	54.24	0
1993	924	267	52.04	55.44	0
1993	925	268	53.19	55.01	0
1993	926	269	54.85	57	0
1993	927	270	56.95	58.86	0
1993	928	271	56.43	59.56	0
1993	929	272	61.5	61.76	0
1993	930	273	62.84	63.24	0

Appendix F. Daily air temperature, soil temperature and percipitation collected by the Campbell Scientific CR-21 Data logger at the Moses Lake, Washington study site.

Month/ Year	Month/ Day	Julian Day	Air Temp. (°F)	Soil Temp. (°F)	Precipitation (inches)
OCTOBER					
1993	1001	274	66.97	65.59	0
1993	1002	275	60.79	63.39	0
1993	1003	276	60.62	63.1	0
1993	1004	277	59.7	62.72	0
1993	1005	278	58.36	62.3	0
1993	1006	279	57.86	60.34	0.11
1993	1007	280	56.75	56.26	0
1993	1008	281	55.58	54.59	0
1993	1009	282	54.16	54.72	0
1993	1010	283	50.77	53.68	0
1993	1011	284	48.21	52.09	0
1993	1012	285	53.63	55.12	0
1993	1013	286	53.24	58.14	0
1993	1014	287	50.17	56.81	0
1993	1015	288	52.1	57.37	0.04
1993	1016	289	48.58	56.55	0
1993	1017	290	44.72	54.82	0
1993	1018	291	49.94	54.27	0
1993	1019	292	46.6	47.81	0
1993	1020	293	47.79	47.96	0
1993	1021	294	44.81	45.65	0
1993	1022	295	46.58	48.01	0
1993	1023	296	47.13	47.35	0
1993	1024	297	58	53.21	0
1993	1025	298	49.18	49.89	0
1993	1026	299	40.51	45.64	0
1993	1027	300	41.34	44.84	0
1993	1028	301	35.94	40.85	0
1993	1029	302	45.53	44.88	0
1993	1030	303	40.84	43.16	0
1993	1031	304	38.63	41.66	0

APPENDIX G. GLP STATEMENTS OF COMPLIANCE FROM COLLINS AGRICULTURAL CONSULTANTS, INC.

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CAC, Inc. No. 11493 V SPONSOR No. 62993 0241-6062-340

STATEMENT OF COMPLIANCE WITH

GOOD LABORATORY PRACTICES

THE DATA SUBMITTED IN THIS NOTEBOOK WAS COLLECTED AND DOCUMENTED ACCORDING TO PRESENT STANDARDS OF GOOD LABORATORY PRACTICES (GLP) 40-CFR PART 160. AND ACCURATELY REFLECT THE RESULTS OF THE TEST. AND ANY AMENDMENTS AND OR DEVIATIONS FROM THE PROTOCOL OR STANDARD OPERATING PROCEDURES HAVE BEEN DOCUMENTED AND REPORTED.

TEST SYSTEM FIELD MANAGER (SIG.) [signature] DATE 12/15/93

QUALITY ASSURANCE UNIT (SIG.) [signature] DATE Jan. 11 1994

COMMENTS: [signature] 12/15/93

PERSONNEL VERIFICATION

I (WE) THE UNDERSIGNED HEREBY DECLARE THAT TO THE BEST OF OUR KNOWLEDGE THE DATA HEREIN IS A TRUE AND ACCURATE RECORD OF THE STUDY. NAMES, SIGNATURES, INITIALS AND DATES OF ALL INDIVIDUALS WHO ENTERED DATA AND/OR WORKED ON THIS TEST SYSTEM:

FULL NAME (PRINT)	FULL NAME (SIG.)	INITIALS	DATE
Vernon Fisher, Jr	[signature]	[signature]	5/24/93
Marie E Bourgeois	[signature]	MEB	6/1/93
Paul J Ilkiff	[signature]	PJI	6-2-93
Margaret Williams	[signature]	MW	1/11/94
	[signature] 12/17/93		

3/93 Rev.

COLLINS AGRICULTURAL CONSULTANTS, INC.

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QUALITY ASSURANCE INSPECTION STATEMENT

Quality Assurance Inspection/Audit Record for the Field Portion of:

CAC, Inc. No. 114936 Sponsor Protocol No. 041293

Sponsor Trial No. 041293/FIFRA 164-1 Amine

Inspection ____ (Y/N): Records ____ Procedure ____ Facilities ____

Audit Y (Y/N): Records Y Procedure N Facilities N

Procedure(s)/Portion(s) that were Audited: Protocol Review

Quality Assurance Signature: Donald L Klotz Date: May 28 1993

Date Reported: Mgt: July 02, 1993 Study Director: July 02, 1993

Inspection Y (Y/N): Records Y Procedure Y Facilities Y

Audit ____ (Y/N): Records ____ Procedure ____ Facilities ____

Procedure(s)/Portion(s) that were Audited: Raw Data review, Calibration of Spray Equipment, Application of Test Substance, Collection of Samples, Fi[illegible]

Quality Assurance Signature: Donald L Klotz Date: June 02, 1993

Date Reported: Mgt: July 02, 1993 Study Director: July 02, 1993

Inspection ____ (Y/N): Records ____ Procedure ____ Facilities ____

Audit Y (Y/N): Records Y Procedure N Facilities N

Procedure(s)/Portion(s) that were Audited: Completed Raw Data review

Quality Assurance Signature: Donald L Klotz Date: Jan 05 1994

Date Reported: Mgt: Jan. 14, 1994 GLK Study Director: Jan. 14 1994 GLK

Quality assurance inspection/audit reports for this study have been submitted to the study director, sponsor management, and CAC, Inc. management.

Quality Assurance Representative (sig.): Donald L Klotz Date: Jan. 14 1994

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CRC, Inc. No. 1193M SPONSOR No. 129830393610630

STATEMENT OF COMPLIANCE WITH

GOOD LABORATORY PRACTICES

THE DATA SUBMITTED IN THIS NOTEBOOK WAS COLLECTED AND DOCUMENTED ACCORDING TO PRESENT STANDARDS OF GOOD LABORATORY PRACTICES (GLP) 40-CFR PART 160. AND ACCURATELY REFLECT THE RESULTS OF THE TEST. AND ANY AMENDMENTS AND OR DEVIATIONS FROM THE PROTOCOL OR STANDARD OPERATING PROCEDURES HAVE BEEN DOCUMENTED AND REPORTED.

TEST SYSTEM FIELD MANAGER (SIG.) _____ DATE 5/ available 4/27/95

QUALITY ASSURANCE UNIT (SIG.) _____ DATE 4/27/95

COMMENTS: _____

PERSONNEL VERIFICATION

I (WE) THE UNDERSIGNED HEREBY DECLARE THAT TO THE BEST OF OUR KNOWLEDGE THE DATA HEREIN IS A TRUE AND ACCURATE RECORD OF THE STUDY. NAMES, SIGNATURES, INITIALS AND DATES OF ALL INDIVIDUALS WHO ENTERED DATA AND/OR WORKED ON THIS TEST SYSTEM:

FULL NAME (PRINT)	FULL NAME (SIG.)	INITIALS	DATE
Timothy L Miller		TLM	5-2-95

3/93 Rev.

COLLINS AGRICULTURAL CONSULTANTS, INC.

QUALITY ASSURANCE INSPECTION STATEMENT

Quality Assurance Inspection/Audit Record for the Field Portion of:

CAC, Inc. No. 11593 M Sponsor Protocol No. 041293/FIFRA 123-1 Amylase

Sponsor Trial No. 12793-0373-6106-360

Inspection ____ (Y/N): Records ____ Procedure ____ Facilities ____

Audit X (Y/N): Records Y Procedure Y Facilities Y

Procedure(s)/Portion(s) that were Audited: Protocol Review, SOP

Quality Assurance Signature: Donald L Klopp Date: June 16, 1993

Date Reported: Mgt: July 02, 1993 SOP Study Director: July 02, 1993 SOP

Inspection X (Y/N): Records Y Procedure Y Facilities Y

Audit ____ (Y/N): Records ____ Procedure ____ Facilities ____

Procedure(s)/Portion(s) that were Audited: Raw Data Interview, Calibration of Sprayer, Application of test substance, Sampling of Soil Core, Plot Size

Quality Assurance Signature: Donald L Klopp Date: June 17, 1993

Date Reported: Mgt: July 02, 1993 Study Director: July 02, 1993

Inspection X (Y/N): Records Y Procedure Y Facilities Y

Audit ____ (Y/N): Records ____ Procedure ____ Facilities ____

Procedure(s)/Portion(s) that were Audited: Raw Data Interview, Sampling of Soil Core, Plot Layout, Calibration, Sample Storage

Quality Assurance Signature: Donald L Klopp Date: SEP. 15 1993

Date Reported: Mgt: OCT. 27, 1993 Study Director: OCT. 27, 1993

Quality assurance inspection/audit reports for this study have been submitted to the study director, sponsor management, and CAC, Inc. management.

Quality Assurance Representative (sig.): Donald L Klopp Date: April 11/1994

COLLINS AGRICULTURAL CONSULTANTS, INC.

QUALITY ASSURANCE INSPECTION STATEMENT

Quality Assurance Inspection/Audit Record for the Field Portion of:

CAC. Inc. No. 11593M Sponsor Protocol No. 041293 / FIFRA 164-1 Amend

Sponsor Trial No. 12993-0393-6106-30

Inspection ____ (Y/N): Records ____ Procedure ____ Facilities ____

Audit X (Y/N): Records Y Procedure ✓ Facilities ✓

Procedure(s)/Portion(s) that were Audited: Completed Raw Data notebook Audit Interim.

Quality Assurance Signature: Donald L Klotz Date: Feb. 09 1994

Date Reported: Mgt: April 11, 1994 Study Director: April 11, 1994

Inspection ____ (Y/N): Records ____ Procedure ____ Facilities ____

Audit Y (Y/N): Records Y Procedure ✓ Facilities ✓

Procedure(s)/Portion(s) that were Audited: Completed Raw Data notebook Audit, Final Audit

Quality Assurance Signature: Donald L Klotz Date: Feb 19, 1994

Date Reported: Mgt: April 11, 1994 Study Director: April 11 1994 (Revised Clarity) ~~April 11 1994~~ DLK 4/11/94

Inspection ____ (Y/N): Records ____ Procedure ____ Facilities ____

Audit ____ (Y/N): Records ____ Procedure ____ Facilities ____

Procedure(s)/Portion(s) that were Audited: DLK April 9, 1994

Quality Assurance Signature: _____ Date: _____

Date Reported: Mgt: _____ Study Director: _____

Quality assurance inspection/audit reports for this study have been submitted to the study director, sponsor management, and CAC, Inc. management.

Quality Assurance Representative (sig.): Donald L Klotz Date: April 11, 1994