Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Thifensulfuron Methyl

MRID #: 418217-01

Matrix: Soil

Analysis: HPLC/PCD

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Du Pont Report No. AMR-1550-89

418217-01

Study Title

Residue Method For Extraction and HPLC Analysis of Harmony® Herbicide From Soil Matrices

Data Requirement

U. S. EPA Pesticide Assessment Guidelines
Subdivision O, 171-4

Authors

Lawrence R. Proksch
Michael P. Wadsley

Study Completed on

November 17, 1989

Report Reformatted on

March 18, 1991

Performing Laboratory

E. I. du Pont de Nemours and Company
Du Pont Agricultural Products
Research and Development Division
Experimental Station
Wilmington, DE 19880-0402

Laboratory Project ID
AMR-1550-89

Statement of No Data Confidentiality Claims

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA Section 10(d), (1)(A), (B), or (C).

Company: E. I. du Pont de Nemours and Company

Company Agent: Priscilla L. Friedman (Typed Name) Date: 3/9/91

Registration Specialist (Title)

Priscilla L. Friedman (Signature)

GOOD LABORATORY PRACTICE STATEMENT

Experimental work performed for this study was conducted in the spirit of EPA Good Laboratory Practice Regulations (40 CFR Part 160).

Sponsor: E. I. du Pont de Nemours and Company

Submitter: E. I. du Pont de Nemours and Company

Authors:

Lawrence R. Proksch
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Date: 18 Mar 91

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Date: 3-18-91

Technical assistance was provided by Dr. J. R. Wheeler and Dr. D. M. Johnson.

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Residue Method For Extraction and HPLC Analysis of Harmony® Herbicide from Soil Matrices

Lawrence R. Proksch and Michael P. Wadsley

INTRODUCTION AND SUMMARY

Scope

An analytical method is described for extraction and analysis of DPX-M6316, the active ingredient in Harmony® Herbicide, in soil with a quantitation limit of 100 parts per trillion (ppt). Both Harmony® Herbicide and DPX-M6316 will be referred to in the text, the term DPX-M6316 referring to the active ingredient in Harmony® Herbicide. The method involves a methylene chloride/acetonitrile extraction, sample concentration and clean-up, followed by reversed-phase HPLC analysis with UV detection at 230 nm. The separation was achieved by column-switching between a phenyl and ODS column and eluent switching between an acidic and basic aqueous mobile phase. This provided an adequately low background for 100-ppt detection of DPX-M6316 in several soil types. The soils were classified as organogenic, sand, and clay.

A validation method using thermospray LC/MS with selective ion monitoring must be incorporated for detection and confirmation of positive results. The same separation approach is applied, however, the UV detection is replaced with the mass spectrometer. The method validation provides adequate qualitative data to minimize possible misinterpretation of the data.

Principles of the Method

The method is an adaptation of the previously submitted Du Pont residue method AMR-1241-88 (EPA MRID# 410826-32, Reference 1). The analysis involves separation using both eluent switching and column switching. Three sequential injections of DPX-M6316 are made on the phenyl column to establish a retention time window for column switching. The column-switching valve is timed to introduce DPX-M6316 onto the ODS column, which is in series with the first column. The valve is switched prior to and after elution of the DPX-M6316 peak.

The DPX-M6316 first elutes from the phenyl column in a basic aqueous mobile phase. Acidified water is then added at a rate of approximately 0.6 mL/min to acidify the aqueous mobile phase containing the DPX-M6316 to a pH of approximately 2.2, well below the pK_a of the sulfonylurea (pK_a=3.5). This weak mobile phase introduces the DPX-M6316 onto the ODS column. The low solvent strength causes the DPX-M6316 to concentrate at the head of the column.

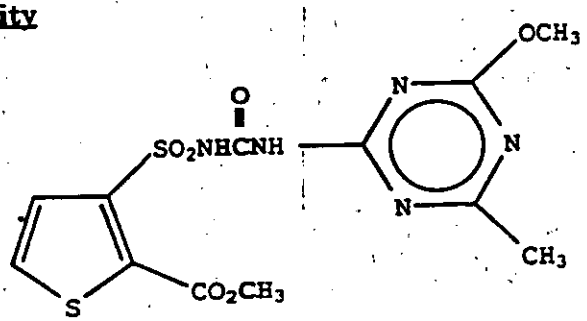
The column-switching valve is then positioned such that the columns are no longer in series. The mobile phase strength is increased to remove any remaining impurities from the phenyl column, and a second switching valve is actuated to divert the flow of mobile phase only to the ODS column for separation and UV detection.

Reproducibility of the DPX-M6316 retention must be monitored by injecting a standard solution after every two sample injections. Because of the complexity of this separation, matrix effects, pH of mobile phase, or even slight changes in pump performance will affect the sulfonylurea retention.

Chromatographic conditions can be optimized to accommodate different background profiles found in various soil samples. Retention times on the phenyl and ODS columns are lengthened by decreasing the organic content of the mobile phase. Also, the retention time is related to the mobile phase pH; slight changes in mobile phase pH can result in relatively large retention time differences and may help resolve DPX-M6316 from a closely eluting impurity.

MATERIALS

Compound Identity



DPX-M6316

Methyl 3-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]-carbonyl]amino]sulfonyl]-2-thiophene carboxylate

Equipment

Chromatograph:	Hewlett Packard 1090M, or other liquid chromatographic system with a ternary pumping system, column oven and a 5-mL injection loop
Metering Pump:	Bodine Electric Company Type NYC-13D3
Detector:	Hewlett Packard 1040A photo-diode array detector, or other variable wavelength UV detector
Switching Valves:	six-port Rheodyne
Columns:	*first column*: Zorbax® phenyl column, 25 cm x 4.6 mm *second column*: Zorbax® ODS column, 15 cm x 4.6 mm
Integrator:	HP 1090M data system
Chromatographic Recorder:	Hewlett Packard Think Jet printer
pH meter:	Beckman Model 44 pH meter
Homogenizer:	Tekmar® Tissumizer® with SDT182EN shaft

Equipment (cont'd)

Rotary Evaporator:	Büchi Model 011 Rotavapor
Filters:	0.45 μm Teflon filters, SM-256 for organic solvents (Bodman Chemical, Aston, PA)

Reagents

Mobile Phase:	Milli-Q® deionized, distilled water, adjusted to pH 3.0 with 85% phosphoric acid (±0.1 pH)
	Milli-Q® deionized, distilled water, adjusted to pH 1.5 with 85% phosphoric acid (±0.1 pH)
	10 mM KH_2PO_4 aqueous buffer (adjusted to pH 7.5 with 80% NaOH (±0.1 pH)
	methanol
Phosphoric Acid:	85% solution, HPLC grade (Fisher Scientific, King of Prussia, PA)
Methanol:	HPLC grade (EM Science, Cherry Hill, NJ)
Methylene Chloride:	HPLC grade (EM Science, Cherry Hill, NJ)
Carbon Tetrachloride:	Reagent grade (EM Science, Cherry Hill, NJ)
Potassium Phosphate:	Monobasic, KH_2PO_4 (J. T. Baker Scientific, Philipsburg, NJ)
Sodium Hydroxide:	50% solution (w/w), "Baker Analyzed" Reagent Grade (J. T. Baker Scientific, Philipsburg, NJ)
Acetonitrile:	HPLC grade (EM Science, Cherry Hill, NJ)

Standards

DPX-M6316-53	98.3% pure analytical standard (E. I. du Pont de Nemours and Co., Du Pont Agricultural Products, Wilmington, Delaware, 19880-0402.

ANALYTICAL METHOD

Sample Preparation and Extraction

The extraction procedure is as follows:

1. Weigh out 400 g of soil.
2. Divide into 50-g portions into 200-mL glass centrifuge jars.
3. Add a known aliquot of water to the soil to ensure a moisture content of 18, 36, and 99%, for sand, clay, and organic soils, respectively. Soil moisture % can be calculated using the equation in Table I.
4. Add 50 mL of acetonitrile and 50 mL of methylene chloride to each 50-g portion of soil.
5. Homogenize with Tissumizer® or a similar homogenizer for two minutes.
6. Centrifuge at 2000 RPM for five minutes.
7. Decant and combine solvent from each jar into a 1000-mL round-bottom flask.
8. Place the round-bottom flask onto a rotary evaporator and partially submerge the flask in a room temperature water bath. Connect the evaporator to a house vacuum to facilitate solvent removal. Evaporate the solvent to a volume of ~1 mL.
9. Rinse the sides of the round-bottom flask thoroughly with 10 mL of methylene chloride to concentrate the extract at the bottom of the flask.
10. Concentrate extract in the round-bottom flask to a volume of ~2 mL under a stream of nitrogen.
11. Quantitatively transfer the extract to a 125-mL glass separatory funnel using two 5-mL portions of carbon tetrachloride to rinse the round bottom flask.
12. Add 10 mL of 10 mM KH_2PO_4 buffer to the separatory funnel. Place the stopper into the separatory funnel and shake vigorously for two minutes. Place the separatory funnel onto a stand and allow the phases to separate.
13. Discard the organic layer.
14. Collect the aqueous layer into a 50-mL centrifuge tube.
15. Centrifuge the aqueous layer at 2000 rpms for five minutes to break any emulsions that form. There may be CCl_4 at the bottom of the tube if an emulsion was present. If so, do not load CCl_4 into the sample locp; load only the aqueous phase. The samples are now ready for HPLC analysis. Note that the analysis can be paused at this point if the samples are kept refrigerated. The HPLC analysis should be performed within 24 hours.

Total sample preparation time is approximately two hours, allowing four samples per technician per day.

RESULTS

Four soil types, differing in pH, organic content, and ionic content, were analyzed. Extraction recoveries for the various soil types are presented in Table I and Table II. The soils in Table I were fortified with ^{14}C-labeled DPX-M6316. The extracts were analyzed by liquid scintillation counting and the extraction recoveries were determined from the net disintegrations per minute. The soil samples described in Table II were fortified with nonradiolabeled DPX-M6316. The extracts of these soil samples were analyzed by HPLC with UV detection. A calibration curve of peak height versus the amount of DPX-M6316 injected was generated and used to quantitate the DPX-M6316 in the soil extracts. Table I provides moisture content and recovery data for each fortification level. Characterization data for each soil type are in Table III.

The chromatographic conditions used for all analyses of DPX-M6316 are listed in Table IV. The configuration of the valve design for column and eluent switching is provided in Figure 1. Figure 2 illustrates how switching times for introduction of the DPX-M6316 onto the ODS column were determined via a sample chromatogram. A sample chromatogram of a DPX-M6316 standard is shown in Figure 3.

Sand, clay, and organogenic soil samples fortified at 100 ppt are shown in Figures 4-6 with superimposed non-fortified samples. The UV profile of the non-fortified sample showed no interference at the retention time of DPX-M6316..

Average extraction recoveries for freshly fortified ^{14}C samples at the 100-ppt level in the various soils are as follows:

Organogenic:	87%
Sand:	87%
Clay:	88%

These values were obtained by extraction of radiolabeled ^{14}C compounds and liquid scintillation counting from the four soil types at several fortification levels. Optimum recoveries of DPX-M6316 were achieved when moisture contents were 18, 36, and 99% for sand, clay, and organic soils, respectively. Average recoveries determined by the HPLC-UV analysis are provided in Table II. Standard calculations are provided in the referenced Du Pont method, AMR-1241-88. Aged samples will be examined in the future.

Standard solutions of DPX-M6316 used to construct a calibration curve are made by introducing aliquots of a 10-µg/mL stock solution into 100-mL volumetric flasks and diluting to volume with water/methanol (90:10, v/v). Figure 8 contains a standard calibration curve for DPX-M6316.

QUALITY CONTROL

At the low levels detected in this method, cross-contamination from various sources in the laboratory will be a problem. To minimize contamination and ensure the integrity of the results, note the following sampling and laboratory techniques:

Sampling Techniques

- Use a decontaminated soil probe with a known inside diameter. The probe must be capable of sampling to the required depth.
- Sample the untreated control soil before the treated soil.
- When sampling the treated soil, sample in order from the soil treated at the lowest rates to the soil treated at the highest rates; or, sample in order from the highest PHI to the lowest.
- Clean trash from the soil surface. Hold the probe perpendicular to the soil surface and insert it to the proper depth. Carefully remove the probe so that soil does not transfer from one depth to another.
- Distinguish the top from the bottom of each tube. Identify each sample with a unique name or number.
- Bag and freeze samples as soon as possible after collection. Close sample bags and probes securely.

Laboratory Techniques

- Conduct the extractions and sample preparation in a laboratory that does not contain DPX-M6316 or any other sulfonylurea.
- Wash all glassware with reagent grade nitric acid and rinse sequentially with hot water, distilled water, methanol, and methylene chloride.
- Keep all glassware covered when possible.
- Do not store glassware in any area where sulfonylurea may be present.
- If fortified samples are to be extracted, weigh and fortify the soil samples with DPX-M6316 in an area other than the area used for the extractions.
- Use a separate Tissumizer® probe to agitate the fortified and non-fortified samples.
- Use clean, properly washed syringes to introduce the samples and standards into the LC; preferably, use a separate syringe for each type of sample.
- Clean the 5-mL sample loop thoroughly before introducing each sample by flushing the loop with 30-mL of the 10 mM KH_2PO_4 buffer. (Sulfonylureas have been known to adhere to stainless steel, thereby affecting the method reproducibility.)

CONCLUSIONS

Quantitation levels of 100 ppt for DPX-M6316 were achieved. The nonradiolabeled recoveries, which are given in Table II, were ≥78% for the 100-ppt fortification level. The average method recoveries were above 80% consistently for all soil types fortified at levels of 100 ppt or higher. Extraction efficiencies were validated using ^{14}C-radiolabeled DPX-M6316 and liquid scintillation counting.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the helpful discussions, information and technical assistance of the following people whose contributions made this method possible: D. M. Johnson, J. R. Wheeler, J. H. Larochelle, L. J. Major, M. J. Sommers, L. Thornton and D. E. Walker.

The original report was reformatted by R. J. Hay and D. M. Johnson.

CERTIFICATION

Residue Method For Extraction and
HPLC Analysis of Harmony® Herbicide from Soil Matrices

We, the undersigned, declare that the work described in this report was performed under our supervision, and that this report, to the best of our knowledge, provides an accurate record of the procedures and results.

Report by:

Lawrence R. Proksch
Laboratory Technician — 18 Mar 91 — Date

Michael P. Wadsley
Laboratory Technician — 3-18-91 — Date

Approved by:

David M. Johnson
Research Supervisor — 3/18/91 — Date

Date Study Completed: November 17, 1989

Date Report Reformatted: March 18, 1991

Storage Location of Raw Data, Specimens, and Final Report:
E. I. du Pont de Nemours and Company
Du Pont Agricultural Products
Experimental Station
Wilmington, Delaware 19880-0402
and/or
Du Pont Records Management Center
Wilmington, Delaware 19880-0870

Sponsor:
E. I. du Pont de Nemours and Company
Du Pont Agricultural Products
Research and Development Division
Experimental Station
Wilmington, Delaware 19880-0402

TABLE I

SOIL EXTRACTION OF DPX-M6316 RADIOLABELED ^{14}C

RESULTS

Soil Type	*Moisture Content*	*Fortification Level*	*Recovery*
Sand	air dried	1 ppb	19%
	18% (water added before spiking)	1 ppb	96%
	18% (water added after spiking)	1 ppb	91%
			%RSD = 0.3
	18% (as received)	0.1 ppb	88%
Clay	36% (as received)	100 ppb	87%
	36% (as received)	10 ppb	81%
	36% (as received)	1 ppb	82%
	16% (water added after spike)	1 ppb	69%
	18% (water added after spike)	1 ppb	73%
	20% (water added after spike)	1 ppb	70%
	24% (water added after spike)	1 ppb	68%
	40% (water added after spike)	1 ppb	80%
	50% (water added after spike	1 ppb	76%
	60% (water added after spike)	1 ppb	77%

TABLE I (cont'd)

SOIL EXTRACTION OF DPX-M6316 RADIOLABELED ^{14}C

RESULTS

Soil Type	*Moisture Content*	*Fortification Level*	*Recovery*
Organogenic	air dried	1 ppb	64%
	59% (as received)	1 ppb	70%
			%RSD = 0.7%
	59% (as received)	0.1 ppb	68%
	79% (water added after spike)	1 ppb	73%
	89% (water added after spike)	1 ppb	83%
	99% (water added after spike)	1 ppb	87%

$$\% \text{ moisture} = \frac{\text{moist soil weight - dry weight}}{\text{dry weight}} \times 100$$

TABLE II
SOIL EXTRACTION OF DPX-M6316
UV RESULTS : 230 nm

Soil Type	*Fortification Level (ppb)*	*Recovery (%)*
Sand	5.0	84
	1.0	99
	1.0	103
	1.0	97
	0.1	80
	0.0	ND
Clay	0.1	114
	0.0	ND
Organogenic	0.1	78
	0.1	80
	0.1	115
	0.0	ND

TABLE III

SOIL CHARACTERIZATION DATA

Harris Laboratories Inc.
[illegible] Peach Street, Box [illegible], Lincoln, NE [illegible]

REPORT OF ANALYSIS

Sample Of: SOIL

Submitted By:
E.I. DU PONT DE NEMOURS
P.O. BOX 80402
SID GOLDBERG-AG
WILMINGTON, DE 19880

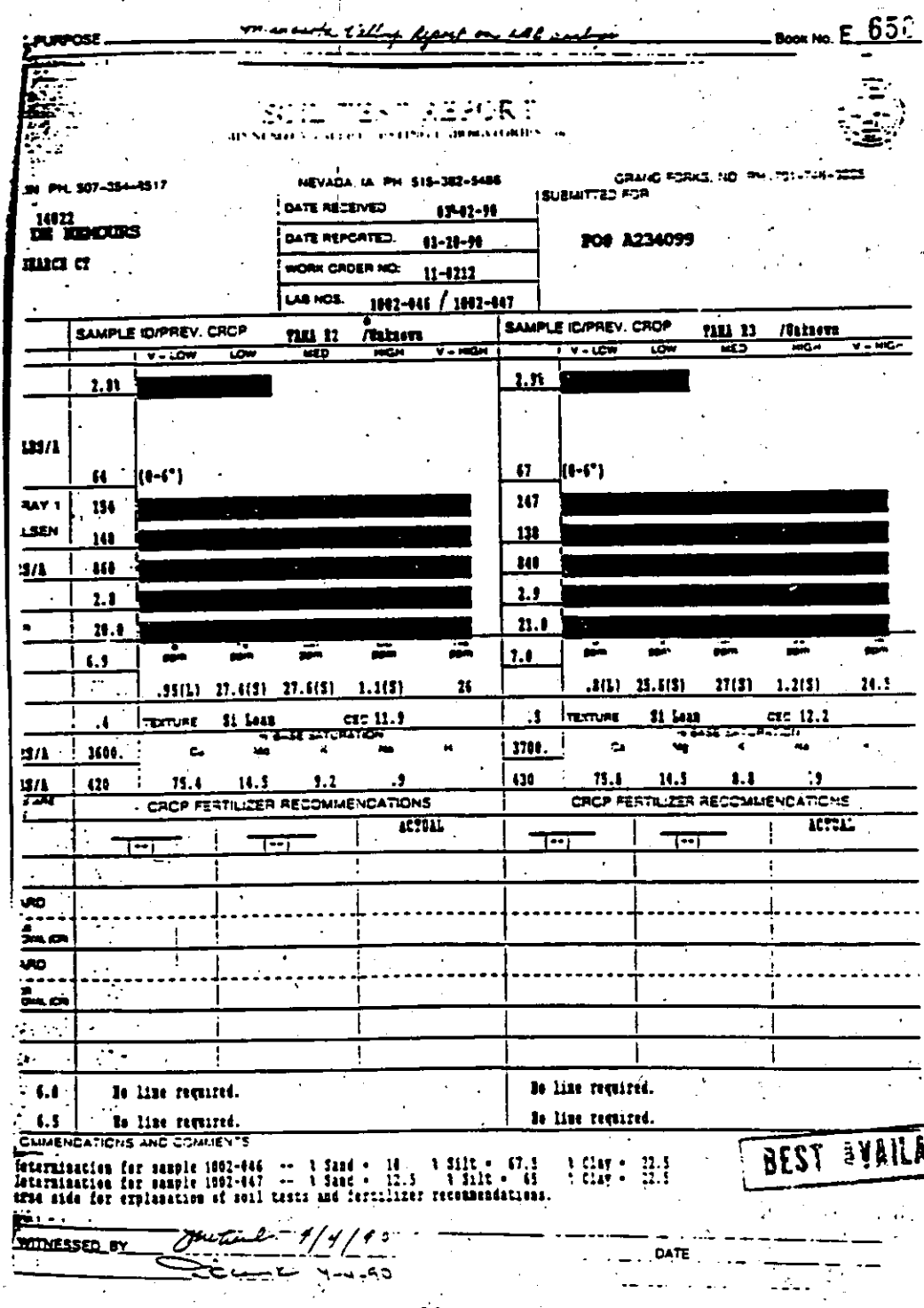
Submitted For:
[illegible] SWEDEN

Date Received	Date Reported	Samples Will Be Stored Until	Laboratory No.
5-OCT-1989	31-OCT-1989	14-NOV-1989	9041855

Client Sample Identification	Analysis	Result
S00071582		
CLAY	% Sand	22.0
	% Clay	50.0
	% Silt	28.0
	Texture	CLAY
	Nitrogen, Total	0.31 %
	Avail. Salts (Saturation)	0.92 MMHOS/CM
	Magnesium	203 PPM
	Potassium	165 PPM
	Calcium	3293 PPM
	Bray 1 Phosphorous	37 PPM
	Organic Matter	2.8 %
	Moisture	26.19 %
	Cation Exchange Capacity	92.68 MEQ/100G
	Holding Cap. (1/3 Bar)	36.40 %
	Holding Cap. (15 Bar)	19.52 %
	pH	6.7
S00071581		
SAND	% Sand	81.6
	% Clay	10.0
	% Silt	8.4
	Texture	LOAMY SAND
	Nitrogen, Total	0.11 %
	Avail. Salts (Saturation)	1.90 MMHOS/CM
	Magnesium	69 PPM
	Potassium	158 PPM
	Calcium	1329 PPM
	Bray 1 Phosphorous	126 PPM
	Organic Matter	1.3 %
	Moisture	1.33 %
	Cation Exchange Capacity	10.01 MEQ/100G
	Holding Cap. (1/3 Bar)	7.09 %
	Holding Cap. (15 Bar)	3.13 %
	pH	INSUFF. SAMPLE
S00071583		
ORGANOGENIC	% Sand	50.4
	% Clay	11.6
	% Silt	38.0
	Texture	LOAM
	Nitrogen, Total	1.34 %
	Avail. Salts (Saturation)	3.60 MMHOS/CM
	Magnesium	153 PPM
	Potassium	105 PPM
	Calcium	1814 PPM
	Bray 1 Phosphorous	29 PPM
	Organic Matter	7.9 %
	Moisture	30.53 %
	Cation Exchange Capacity	116.85 MEQ/100G
	Holding Cap. (1/3 Bar)	77.74 %
	Holding Cap. (15 Bar)	35.59 %

TABLE III (cont'd)

SOIL CHARACTERIZATION DATA



PURPOSE ... Book No. E 650

SOIL TEST REPORT

... PH 507-354-8517 | NEVADA, IA PH 515-382-5486 | GRAND FORKS, ND PH ...

14022
DE HEMOURS
SEARCH CT

DATE RECEIVED 03-02-90
DATE REPORTED 03-20-90
WORK ORDER NO: 11-0212
LAB NOS. 1002-046 / 1002-047

SUBMITTED FOR
PO# A234099

	SAMPLE ID/PREV. CROP	FIELD 12 /Unknown					SAMPLE ID/PREV. CROP	FIELD 13 /Unknown				
		V-LOW	LOW	MED	HIGH	V-HIGH		V-LOW	LOW	MED	HIGH	V-HIGH
	2.91						3.95					
LBS/A	64	(0-6")					67	(0-6")				
MAY 1	156						247					
LSEN	148						138					
S/A	860						840					
	2.8						2.9					
	20.0						21.0					
	6.9						7.0					
		.95(L)	27.6(S)	27.6(S)	1.1(S)	26		.8(L)	25.5(S)	27(S)	1.2(S)	24.5
	.4	TEXTURE	SL Loam		CEC 11.9		.5	TEXTURE	SL Loam		CEC 12.2	
S/A	3600.	% BASE SATURATION Ca	Mg	K	Na	H	3780.	% BASE SATURATION Ca	Mg	K	Na	H
S/A	420	75.4	14.5	9.2	.9		430	75.8	14.5	8.8	.9	
ZONE	CROP FERTILIZER RECOMMENDATIONS				ACTUAL		CROP FERTILIZER RECOMMENDATIONS				ACTUAL	
		--	--					--	--			
URD												
ORMULATION												
URD												
ORMULATION												
	6.0	No Lime required.					No Lime required.					
	6.5	No Lime required.					No Lime required.					

COMMENDATIONS AND COMMENTS

Determination for sample 1002-046 -- % Sand = 10 % Silt = 67.5 % Clay = 22.5
Determination for sample 1002-047 -- % Sand = 12.5 % Silt = 65 % Clay = 22.5
Reverse side for explanation of soil tests and fertilizer recommendations.

WITNESSED BY ... DATE ...

TABLE IV

LIQUID CHROMATOGRAPH INITIAL PARAMETERS

Flow: 1.400 mL/min.
Solvent A: 95.0% pH 3. H_2O
B: 0.0% pH 7.5 10 mM KH_2PO_4
C: 5.0% Methanol
Oven Temp.: 40.0°C
Max Pressure: 400 bar

Stop Time: 65.00 min.
Post Time: 0.00 min.
Injection Volume: 0.0 µL
Min. Prassure: off
Column Switch: 0
Contacts: 0000
Slowdown: 2

LIQUID CHROMATOGRAPH TIMETABLE

Time (min.)		*A, 1, 2*	*B*	*C*
0.01	Solvent (%)	95.0	0.0	5.0
0.02	Contact	on		
7.00	Contact	off		
7.01	Solvent (%)	95.0	0.0	5.0
7.02	Solvent (%)	45.0	0.0	55.0
16.90	Solvent (%)	45.0	0.0	55.0
17.00	Solvent (%)	0.0	80.0	20.0
23.50	Flow (mL/min.)	1.400		
23.60	Flow (mL/min.)	1.000		
24.00	Column	1		
29.00	Column	0		
29.10	Flow (mL/min.)	1.000		
29.20	Flow (mL/min.)	1.400		
29.20	Solvent(%)	0.0	80.0	20.0

TABLE IV (Cont'd.)
LIQUID CHROMATOGRAPH TIMETABLE

Time (min.)		*A, 1, 2*	*B*	*C*
30.00	Solvent(%)	20.0	0.0	80.0
35.00	Solvent(%)	20.0	0.0	40.0
40.00	Solvent(%)	60.0	0.0	40.0
42.00	Contact	on		
65.00	Contact	off		
65.10	Solvent (%)	60.0	0.0	40.0
70.20	Solvent (%)	95.0	0.0	5.0

FIGURE 1

VALVE DESIGN FOR COLUMN AND ELUENT SWITCHING

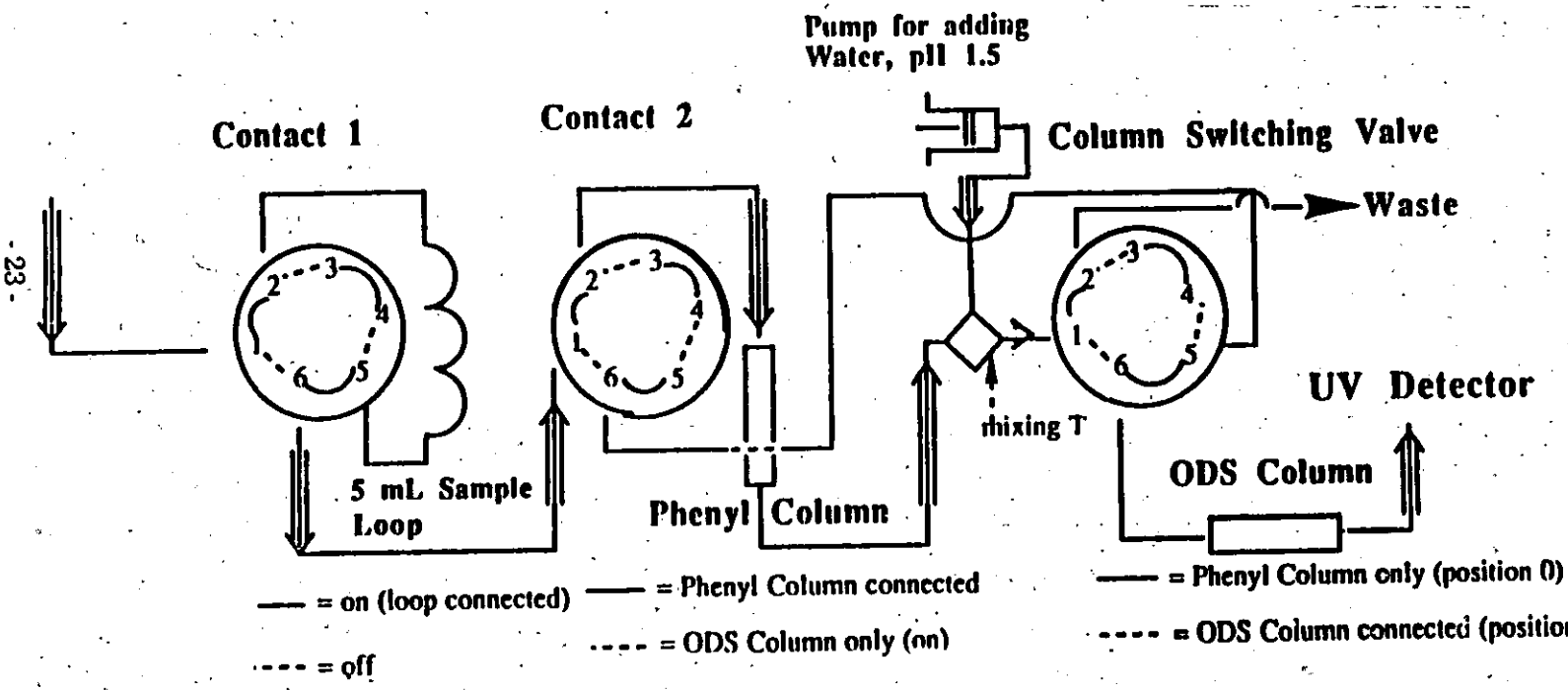


FIGURE 2

ILLUSTRATIVE SWITCHING TIMES FOR DPX-M6316 INTRODUCTION ONTO THE ODS COLUMN

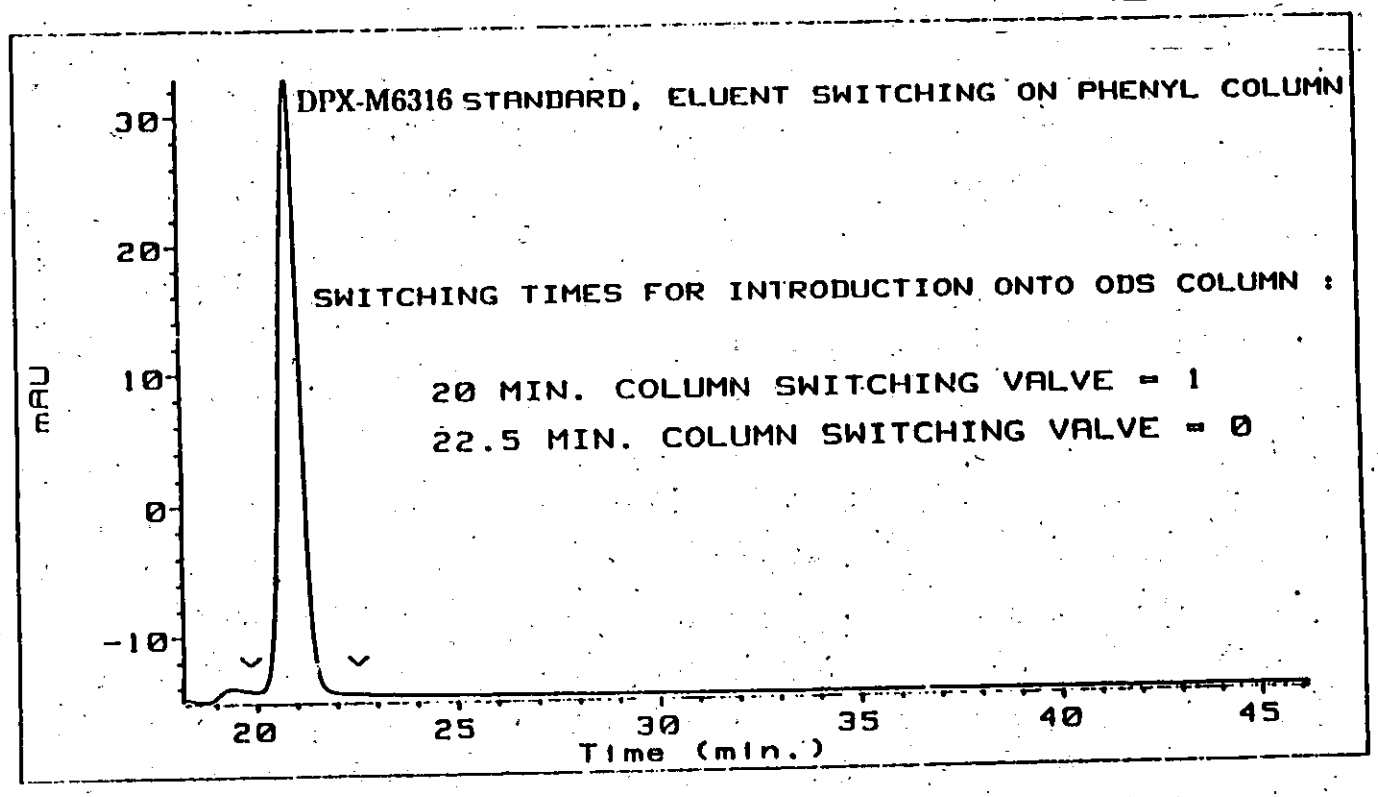


FIGURE 3

TYPICAL CHROMATOGRAM OF A DPX-M6316 STANDARD

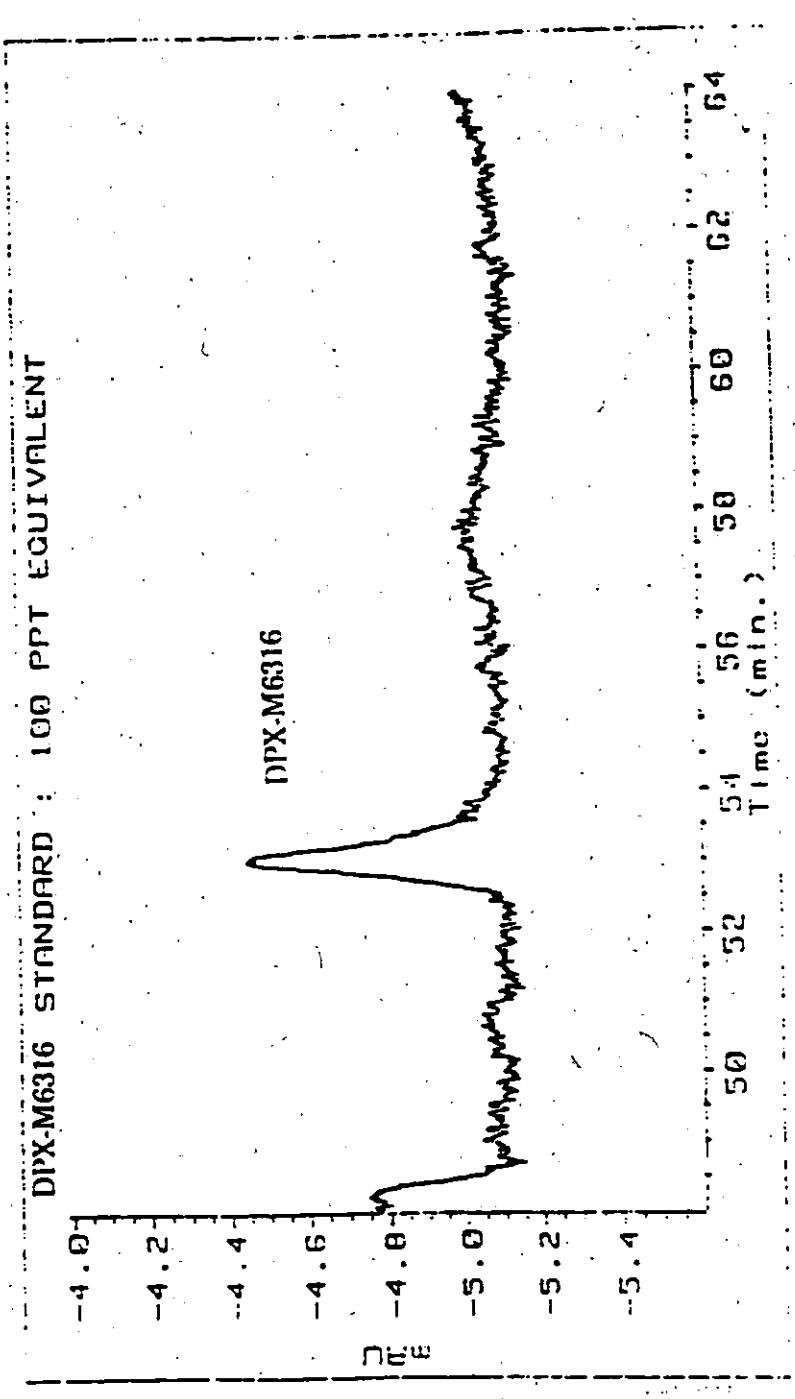


FIGURE 4

TYPICAL CHROMATOGRAM OF SAND
SOIL FORTIFIED WITH 100 PPT OF DPX-M6316

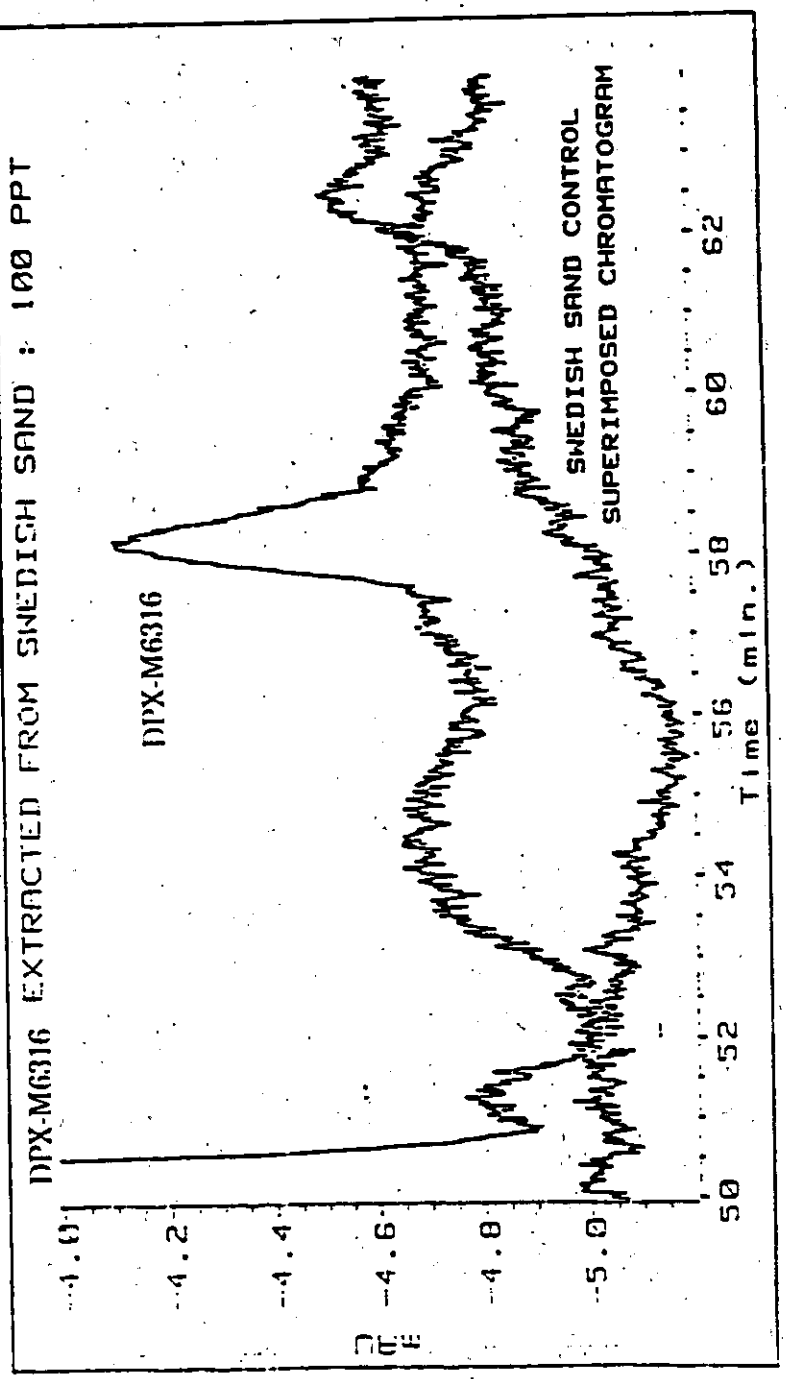


FIGURE 5

TYPICAL CHROMATOGRAM OF CLAY
SOIL FORTIFIED WITH 100 PPT OF DPX-M6316

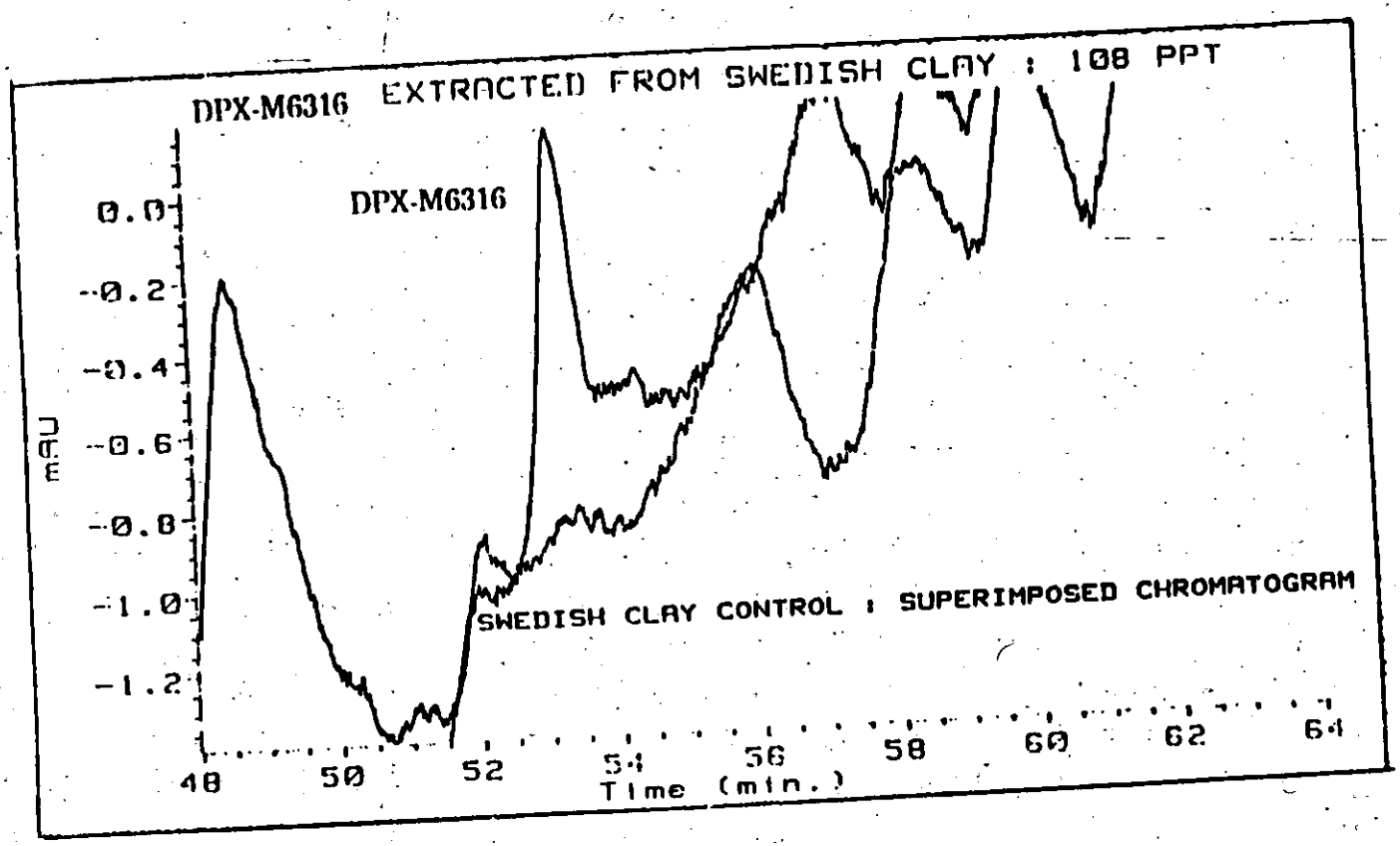


FIGURE 6

TYPICAL CHROMATOGRAM OF
ORGANOGENIC SOIL FORTIFIED WITH 100 PPT OF DPX-M6316

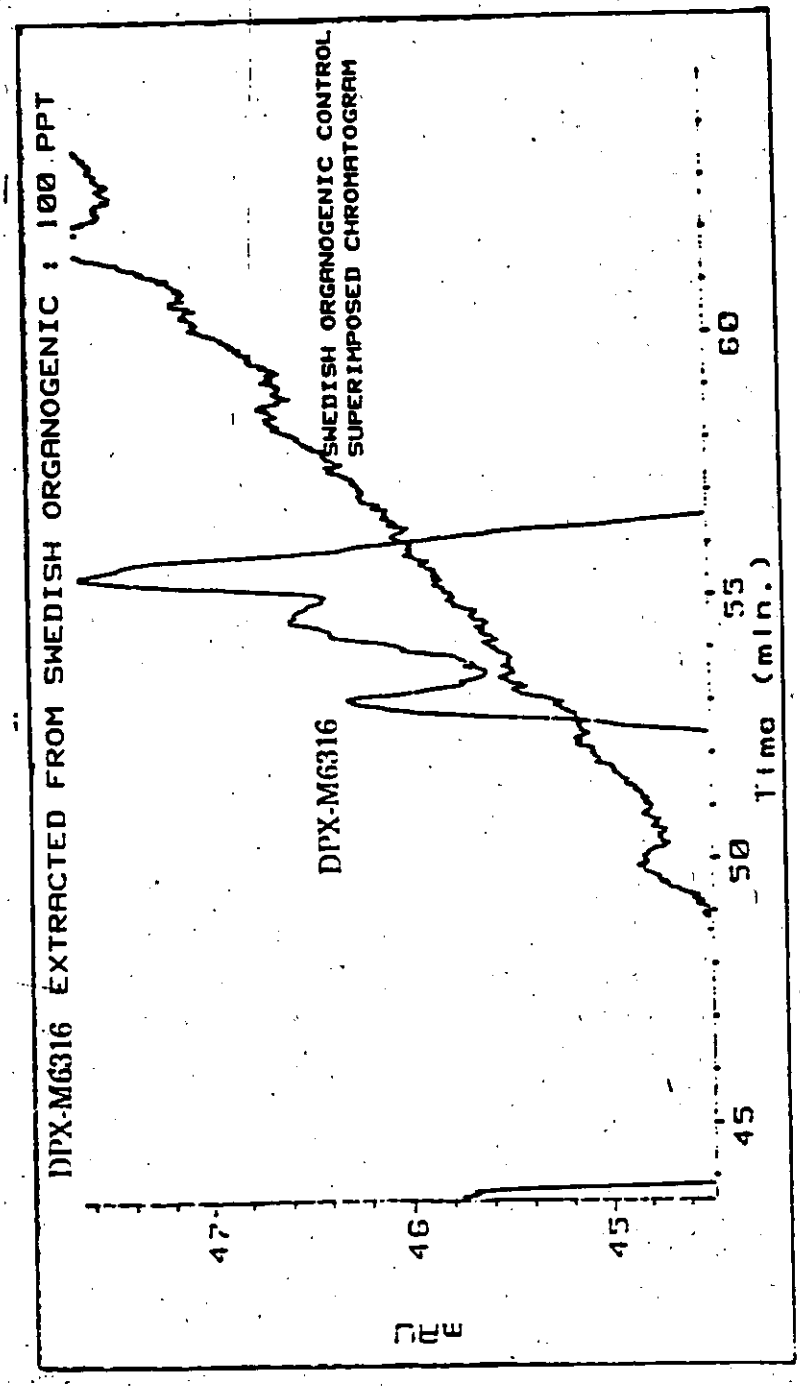
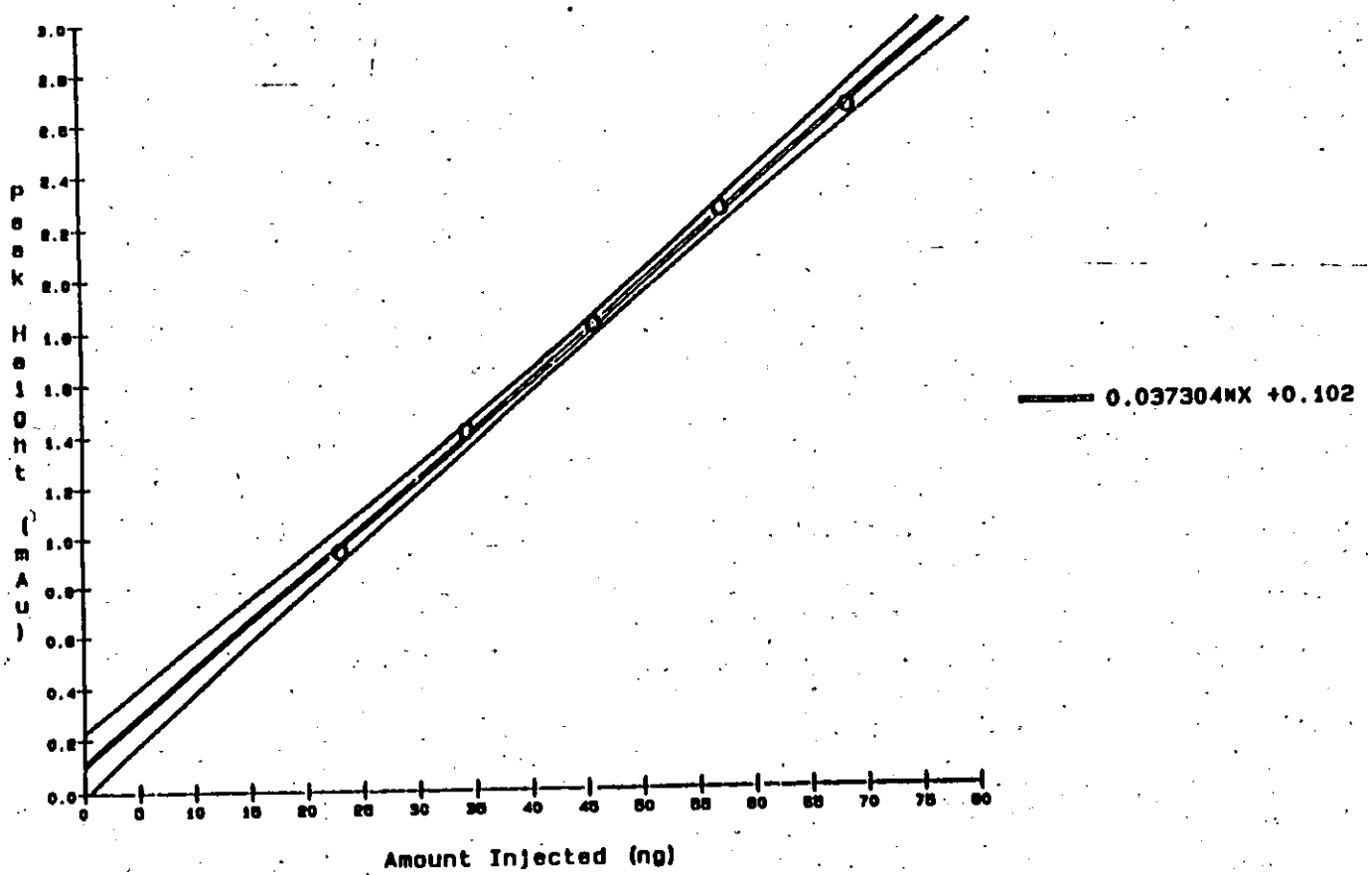


FIGURE 7

STANDARD CALIBRATION CURVE FOR DPX-M6316



[1] The dotted assymptotic line represents the 95% confidence values.

REFERENCES

1. Analytical Method for the Quantitation of DPX-E9636 in Corn (Forage and Grain), Du Pont Report No. AMR-1241-88, J. H. Larochelle, L. J. Major and P. G. Rossi, May, 1989, MRID # 410826-32.