



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

HED 5


OPP OFFICIAL RECORD
HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361


OFFICE OF
PREVENTION, PESTICIDES, AND
TOXIC SUBSTANCES

MEMORANDUM

DATE: October 5, 1999

SUBJECT: PP# 8F04948. DPX MP062 in/on Brassica, Sweet Corn, Cotton. Fruiting Vegetables, Lettuce (Head and Leaf), Pome Fruits, and Animal Commodities. **Results of Petition Method Validations (PMVs)**. MRID#s 44477327, 44477332, and 44477338. Barcode D257972. Chemical No.: 067710. Case: 289487. Submission: S539237.

FROM: Susie Chun, Chemist 
RAB1/HED (7509C)

THROUGH: George Kramer, Ph.D., Chemist 
Melba Morrow, Branch Senior Scientist
RAB1/HED (7509C)

TO: Dan Peacock/Arnold Layne (PM 3)
Registration Division (7505C)

E.I. du Pont de Nemours and Company has submitted a petition for the establishment of tolerances for residues of a new insecticide, DPX-MP062 (proposed common name indoxacarb), in/on various crop commodities, meat, and milk.

DPX-MP062 [(R,S)-methyl 7-chloro-2,5-dihydro-2-[[methoxycarbonyl][4-(trifluoromethoxy)phenyl]amino]carbonyl]indeno[1,2-e][1,3,4]oxadiazine-4a(3H)-carboxylate] is a 75:25 mixture of two enantiomers: DPX-KN128, which is insecticidally active, and [N-KN127 (alternatively identified by the petitioner as DPX-KN127), which is insecticidally inactive. The petitioner is proposing the establishment of tolerances for residues of DPX-MP062 in/on the following raw agricultural commodities (RACs):

Apple, pomace (wet)	6.0 ppm
Pome fruit	2.0 ppm
Head & Stem Brassicas	10.0 ppm
Cottonseed	3.0 ppm
Cotton gin trash	15.0 ppm
Leaf lettuce	20.0 ppm
Head lettuce	7.0 ppm
Fruiting vegetables	0.70 ppm
Sweet corn kernel	0.02 ppm
Sweet corn forage	20.0 ppm
Sweet corn stover	25.0 ppm

Meat	0.02 ppm
Milk	0.10 ppm
Cattle kidney	0.05 ppm

The petitioner is also proposing the establishment of tolerances for residues of the DPX-MP062 active ingredient, DPX-KN128 [(S)-methyl 7-chloro-2,5-dihydro-2-[[methoxycarbonyl][4-(trifluoromethoxy)phenyl]amino]carbonyl]indeno[1,2-e][1,3,4]oxadiazine-4a(3H)-carboxylate], and its metabolite, IN-JT333 [methyl 7-chloro-2,5-dihydro-2-[[4-(trifluoromethoxy)phenyl]amino]carbonyl]indeno[1,2-e][1,3,4]oxadiazine-4a(3H)-carboxylate], as follows:

Milk fat	0.75 ppm
Cattle fat	0.75 ppm

The Health Effects Division (HED) requested that the Analytical Chemistry Laboratory Branch (ACLB) perform petition method validations (PMVs) on the following methods (Memo. D245242, N. Dodd, 4/27/98):

Analytical Enforcement Method (HPLC/Column Switching/UV) for the Determination of Residues of DPX-KN128 and IN-KN127 in Crops", DuPont Report #AMR 2712-93, 08-AUG-1997, MRID#444773-27

Analytical Enforcement Procedure for the Analysis of DPX-KN128/DPX-KN127 in Crops and Related Process Fractions by GC-MSD", DuPont Report #AMR 3493-95, Supplement #2, Revision #1, 02-DEC-1997, MRID #44477332

Analytical Method (HPLC/Column Switching/UV) for the Determination of Residues of DPX-KN128/DPX-KN127 and IN-JT333 in Animal Matrices- Whole and Skim Milk, Cream, Fat, Muscle, Liver, and Kidney, DuPont Report #AMR 3337-95, 11-MAR-1997, MRID #44477338

The results of the PMVs are appended to this memorandum as Attachment 1.

Results Summary

Plant RACs

Method AMR 2712-93

The average recoveries and limit of quantitations (LOQs) are summarized in Table 1. One chemist can prepare six samples for HPLC analysis in 4-6 hours. Instrumental analysis time for duplicate injection of samples and standards is 20-24 hours (unattended and excluding reporting time). Samples were fortified with DPX-MP062. The complete PMV by ACLB is attached (Attachment 3).

Table 1 - ACLB Recoveries and LOQ of Method AMR 2712-93			
Raw Agricultural Commodity (RAC)	Fortification Level (ppm)	% Recovery ^a	ACLB LOQ (ppm)
Apple	0.010	102.8 ± 22.9	0.01
	2.00	89.2 ± 0.4	
Broccoli	0.010	66.6 ± 1.1	0.01
	10.0	66.6 ± 1.3	
Cottonseed	0.010	102.5 ± 3.5	0.01
	3.00	105 ± 1.4	
Cotton Hulls	0.010	89.8 ± 1.2	0.01
	3.00	101.6 ± 9.0	
Cotton Oil	0.010	82.6 ± 4.2	0.01
	3.00	111.5 ± 20.5	
Head lettuce	0.010	93.2 ± 3.2	0.01
	7.00	91.8 ± 1.2	
Tomato	0.01	81.4 ± 13.8	0.01
	0.700	77.8 ± 5.4	
Sweet corn, kernel and cobs with husk removed (K+CWHR)	0.010	120 ± 9.9	0.01
	0.020	74.2 ± 17.3	
Sweet Corn, Forage	0.010	86.8 ± 3.6	0.01
	20.0	93.8 ± 2.5	
Sweet Corn, Stover	0.050	85.1 ± 5.5	0.05
	25.0	65.6 ± 3.4	

^a Each listed value is the mean recovery ± the sample standard deviation based on replicated determinations.

Method AMR 3493-95

The average recoveries are summarized in Table 2. One chemist can prepare and analyze six samples with GC analysis (with an autosampler) in 24 hours. ACLB calculated LOQs and limit of detections (LODs) for only select commodities, which are also shown in Table 2. Samples were fortified with DPX-MP062. The complete PMV ACLB is attached (Attachment 2).

Table 2 - ACLB Recoveries and LOQ of Method AMR 3493-95				
Raw Agricultural Commodity (RAC)	Fortification Level (ppm)	% Recovery ^a	ACLB estimated LOD ^b (ppm)	ACLB calculated LOQ ^c (ppm)
Apple	0.21	150 ± 2.8	0.06	0.18
	2.1	136 ± 2.8		
Broccoli	0.21	102.5 ± 10.6	0.02	0.06
	10.3	95.5 ± 0.7		
Cottonseed	0.21	85.5 ± 13.4	-----	-----
	3.1	93.5 ± 4.9		
Cotton Hulls	0.21	104.5 ± 20.5	-----	-----
	3.1	96.5 ± 0.7		
Cotton Oil	0.21	78.5 ± 3.5	-----	-----
	3.1	71.5 ± 3.5		
Head lettuce	0.21	123.5 ± 13.4	0.01	0.03
	7.2	96.5 ± 9.2		
Tomato	0.21	116.5 ± 3.5	0.03	0.09
	0.72	147.5 ± 12.0		
Sweet corn K+CWHR	0.21	119 ± 7.1	-----	-----
	0.41	108.5 ± 4.9		
Sweet Corn. Forage	0.21	133 ± 26.9	-----	-----
	20.1	84.5 ± 7.8		
Sweet Corn, Stover	0.21	152 ± 26.9	-----	-----
	24.8	90.5 ± 9.2		

^a Each listed value is the mean recovery ± the sample standard deviation based on replicated determinations.

^b LOD estimated as 1/3 calculated LOQ.

^c LOQ calculated as 10 times noise measure peak to peak at area near the retention time of DPX-MP062.

Animal Commodities

The animal method is designated as AMR 3337-95. The average recovery of DPX-MP62 in whole milk was 94 ± 5% and in cattle liver was 80 ± 13%. The average recovery in cattle fat was: 101 ± 7% of DPX-KN128 and 93 ± 8% of the metabolite, IN-JT333. The average recovery in milk cream fat was: 87 ± 7% of DPX-KN128 and 76 ± 13% of the metabolite, IN-JT333. One chemist can prepare eight samples for HPLC analysis in ~16 hours. Instrumental analysis time for duplicate injection of samples and standards is ~28 hours (unattended and excluding

reporting time). The complete PMV ACLB is attached (Attachment 1).

Conclusions

1. The petitioner is requested to submit standards of DPX-MP062, DPX-KN128, and IN-JT333 to the EPA repository.

Plant RACs

Method AMR 2712-93

- 2a. The recoveries are acceptable. The LOQ of 0.01 ppm is applicable to pome fruit, head and stem Brassicas, cotton (seed, forage, hulls, meal, and refined oil), head and leaf lettuce, fruiting vegetables, and sweet corn (K+CWHR) and sweet corn forage. The LOQ of 0.05 ppm is applicable to cotton gin trash and sweet corn stover. ACLB estimated the approximate LOD to be 0.0025 ppm for the commodities investigated. No revisions are recommended by ACLB (Attachment 3).

Method AMR 3493-95

- 3a. The method was found unacceptable for enforcement purposes due to lack of clear instructions in the procedure. Also, there were unacceptable recoveries (>120%) for some RACs. It was recommended by ACLB (Memo, D. Rains and D. Swineford, 9/20/99) not to forward the method to the FDA and to consider the conclusions reported in their memo when revising the method (Attachment 2).
- 3b. The following comments were made by ACLB in the PMV results (Memo, D. Rains, and D. Swineford, 9/20/99).
 - i. Comments on silanizations of glassware should be rewritten in the method to note that DPX-MP062 can adhere to glassware in the absence of organic solvent. The method should be modified to state that glassware **should** be silanized.
 - ii. There were problems with matching the petitioner's claimed sensitivity. The method should be rewritten to include this information concerning no reproducibility of sensitivity.
 - iii. ACLB calculated LOQ's for select commodities and concurs with the petitioner's proposed LOQ of 0.2 ppm as a practical LOQ, due to problems with matrix enhancement.

Animal Commodities

4. The recoveries of DPX-MP062 and its metabolite IN-JT333 are acceptable. ACLB considers this method suitable for food tolerance enforcement. The revisions recommended in the addendum to the ACLB results memo (Memo, P. Golden and P. Schermerhorn, 7/21/99) should be incorporated in the method AMR 33337-95. ACLB estimated the LOD for DPX-MP062 to be 0.002 ppm in whole milk and liver. ACLB estimated the LOD for DPX-KN128 to be 0.003 ppm in both cattle fat and milk cream. ACLB estimated the LOD

for IN-JT333 to be 0.003 ppm in cattle fat and 0.004 ppm in milk cream (milk fat) (Attachment 1).

Recommendations

The animal method and the EPA addendum will be forwarded to FDA for inclusion in PAMII. Any copy of the animal method that is distributed prior to publication in PAM II should include the EPA addendum. The plant method, Method AMR 2712-93, will be forwarded to FDA for inclusion in PAMII. The plant method, Method 3493-95 was found unacceptable for enforcement purposes. Though revisions were recommended, the petitioner does not have to submit a revised method because an adequate plant enforcement method has been submitted (Method AMR 2712-92). **Until the receipt of the standards, the requirements for analytical enforcement methodology will remain unfulfilled.**

Attachment 1- Animal Method AMR 3337-95, Memo, P. Golden and P. Schermerhorn, 7/21/99

Attachment 2 - Plant Method AMR 3493-95: Memo, D. Rains and D. Swineford, 9/20/99

Attachment 3 - Plant Method AMR 2712-93: Memo, E. Hayes, 7/21/99

cc (with attachments): PP#8F4948, S. Chun, F. Griffith (BEAD, Mailcode 7503W)
RDI: M. Morrow (10/6/99), RAB1 Chemists (9/30/99), G. Kramer (9/21/99)
S. Chun:806R:CM#2:(703)305-2249:7509C:(RAB1)

ATTACHMENT 1



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ENVIRONMENTAL SCIENCE CENTER
701 MAPES ROAD
FORT MEADE, MD 20755-5350
Analytical Chemistry Branch

MEMORANDUM

SUBJECT: PP#8F04948. Tolerance Method Validation of DPX-MP062 on Cattle
Liver, Whole Milk, Cattle Fat and Milk Cream
(MRID#:444773-38) ACB #B98-(47-50)

FROM: Paul E. Golden, Chemist
Patricia G. Schermerhorn, Chemist
Analytical Chemistry Branch

THRU: Francis D. Griffith, Jr., Chief,
Analytical Chemistry Branch

THRU: Donald A. Marlow, Laboratory Coordinator
Biological and Economic Analysis Division (7503C)

TO: Karen Whitby, Chief
Registration Action Branch-I
Health Effects Division (7509C)
and
Arnold Layne, Chief
Insecticide Branch
Registration Division (7505C)

INTRODUCTION

The Analytical Chemistry Branch (ACB) was requested by the Registration Action Branch-I (RABI) to conduct a method validation for DPX-MP062 and its metabolite IN-JT333. DPX-MP062 is composed of two enantiomers, DPX-KN128 ("S"-isomer) which is insecticidally active and DPX-KN127 ("R"-isomer) which is insecticidally inactive. IN-JT333 is the primary metabolite of DPX-MP062. Samples were requested to be run in duplicate on liver samples fortified with DPX-MP062 at 0.00 ppm, 0.01 ppm, and 0.02 ppm; milk samples fortified with DPX-MP062 at 0.00 ppm, 0.01 ppm, and 0.10 ppm; and milk fat and cattle fat samples fortified with the "S" isomer and IN-JT333 metabolite each at 0.00 ppm, 0.01 ppm, and 0.75 ppm. ACB contacted HED for clarification concerning the request for 'milk fat'. The method was written to include milk cream and there was no data provided for milk fat by either the registrant or the ILV. Additionally there was no method provided for extracting milk fat from whole milk.

HED agreed to use milk cream in place of milk fat because milk cream represents a high percentage of milk fat. The proposed DPX-MP062 tolerances are 0.02 ppm in/on meat, and 0.10 ppm in/on milk. The proposed "S" isomer and IN-JT333 tolerances are each 0.75 ppm in/on cattle fat and milk fat.

RECOMMENDATIONS

1. ACB finds this LC method suitable for the enforcement of tolerances for DPX-MP062 and the IN-JT333 metabolite in/on cattle liver, whole milk, cattle fat and milk cream. The minor changes recommended in the addendum should be incorporated into the method before use.

2. ACB recommends that the residue analytical method be made available to federal and state enforcement laboratories along with our addendum. ACB recommends that the method be forwarded to FDA's Technical Editing Group in a future revision to PAM-II with a Roman numeral designation.

3. ACB recommends that as a condition of registration the petitioner, DuPont, be required to supply the appropriate amounts of DPX-MP062, DPX-KN128 and IN-JT333 to the National Pesticid Standards Repository at Fort Meade, MD.

METHOD SUMMARY

The analytical method validated from E.I. DuPont de Nemours and Company was titled: "Analytical Method (HPLC/Column Switching/UV) for the Determination of Residues of DPX-KN128/DPX-KN127 and IN-JT333 in Animal Matrices- Whole and Skim Milk, Cream, Fat, Muscle, Liver, and Kidney" by Jennifer S. Amoo and Ellen Beaver-Stetser, coded DuPont Report # AMR 3337-95, and dated 3/11/97 (MRID # 444773-38).

The combined DPX-MP062 enantiomers are analyzed in animal matrices as a single chromatographic peak and the IN-JT333 metabolite is analyzed as a separate distinct peak on non-chiral liquid chromatography columns. The two analytes are extracted from 5-gram aliquots of samples into acetonitrile for milk, cream, and liver and into ethyl acetate for cattle fat samples. Milk and cream samples can be cleaned up using C18 Sep-Pak cartridges if interferences are present (ACB used the C18 clean up during this validation). Fat and liver samples are cleaned up by homogenization and partitioning with hexane. Liver samples require an additional clean-up step involving Silica solid phase extraction. The samples are then analyzed by column-switching HPLC with UV-absorbance detection at 310 nm.

INDEPENDENT LABORATORY VALIDATION (ILV) DATA

ILV data were generated by EN-CAS Analytical Laboratories of Winston-Salem, North Carolina. (see MRID # 444773-39). Whole milk samples were fortified with: DPX-MP062 and IN-JT333 each at 0.01 ppm, and 0.02 ppm. Ground beef samples were fortified with DPX-MP062 and IN-JT333 each at 0.01 ppm, and 0.02 ppm. Samples were then analyzed by the petitioners proposed enforcement method. The initial ground beef trial was successful and the initial whole milk trial was unsuccessful. The method specifies reconstituting the samples in 50 % acetonitrile/50 % KH₂PO₄ buffer, pH 3.0 for sample analysis. After a minor modification to reconstitute the samples in acetonitrile, prior to adding buffer and bringing to final volume for analysis, the second whole milk trial was successful. Recoveries of DPX-MP062 in ground beef ranged from 100% to 113% averaging $108\% \pm 5.8\%$, n=4. Recoveries of IN-JT333 in ground beef ranged from 82% to 89% averaging $85\% \pm 3.0\%$, n=4. Recoveries of DPX-MP062 in the second trial for whole milk ranged from 76% to 85% averaging $81\% \pm 4.1\%$, n=4. Recoveries of IN-JT333 in the second trial for whole milk ranged from 69% to 93% averaging $81\% \pm 11\%$, n=4. The ILV lab concluded that reconstituting first with 100% acetonitrile, vortexing, sonicating and then adding the buffer to the vial appears to improve recoveries in milk. The ILV also concludes that vortexing and sonication are very critical to the success of the method. ACB concludes the ILV data presented for whole milk and ground beef are acceptable.

CONCLUSIONS

1. The DPX-MP062, DPX-KN128 and IN-JT333 analytical standards used for this method validation were supplied by DuPont Ag Products. These standards were not available from the EPA Repository as of 6/17/99. The unused portion of the standards will be retained by ACB in the National Pesticide Standard Repository.

2. The whole milk used for this method validation was obtained from the USDA Milking Parlor. Milk cream was separated from the whole milk by centrifugation at - 4 ° C. Beef fat and liver commodities were obtained from a local grocery store.

3. ACB's results are tabulated on pages 5,6 and 7. Successful trials were made on the first attempt for beef liver, beef fat and milk cream. A second trial was required for whole milk due to the presence of a contaminant in the first trial. The source of the contaminant was traced to the filters used by ACB for final sample filtration just prior to HPLC analysis. ACB found that pre-washing the filters with the final solvent eliminated the contaminant and allowed a successful trial on whole milk. The filter pre-wash was then performed for the liver, beef fat and milk cream trials. ACB concludes there has been a successful TMV for milk, liver, beef fat and milk cream following the method as written with minor modifications and substitutions (see Addendum notes # 1,2,3 & 4).

4. The chromatographic data generated by the ACB was found to be comparable to that submitted by both the registrant and the ILV.

5. The petitioner established the limit of quantitation at 0.01 ppm for DPX-KN128/DPX-KN127 and IN-JT333 in milk, cream and all animal tissues by this HPLC-UV method for each analyte. ACB confirmed the petitioner's LOQ on the commodities tested by ACB. ACB estimated the LOD for DPX-MP062 to be 0.002 ppm in whole milk and liver. ACB's estimated LOD for DPX-KN128 is 0.003 ppm in both cattle fat and milk cream. ACB's estimated LOD for IN-JT333 is 0.003 ppm in cattle fat and 0.004 ppm in milk cream.

6a. ACB concludes that this method meets the requirements for an enforcement method as defined in the Pesticide Test Guidelines, 860.1340. Any copy of the method that is distributed prior to publication in PAM II should include the EPA addendum.

6b. ACB concludes that this method is suitable to gather residue data from the confirmed LOQ to the upper limit tested.

7. Adequate ILV data were presented.

8. ACB concludes that a set of eight (6) samples took one chemist ~16 hours to work up for HPLC analysis. Unattended, duplicate injection of samples and standards required ~ 28 hours to analyze excluding reporting time.

Attachments: Method Validation Results, EPA Addendum, Detector Response

cc: TMV File B98-(47-50); Analysts-PEGolden, PGSchermerhorn; NDodd(HED/RABI, 7509C).
ACB:7503C:Fort Meade ESC. PGolden:6/10/99:(410)305-2960;edit:PGolden:6/23/99.
RDI:QAPanel:6/23/99; TMLDR:MWLaw 6/23/99; BrCh:FDGriffith:7/8/99

Analytical Chemistry Branch
METHOD VALIDATION RESULTS
 PP#8F04948

Commodity	Chemical Added	PPM Added	PPM Found	Percent Recovery
Whole Milk	DPX-MP062	0.000	N.D. ¹	-
		0.000	N.D. ¹	-
		0.010	0.0093	93.0
		0.010	0.0096	96.0
		0.100	0.1010	101
		0.100	0.0876	87.6

¹ N.D.: non-detected (less than 0.002 ppm)

Commodity	Chemical Added	PPM Added	PPM Found	Percent Recovery
Cattle Liver	DPX-MP062	0.000	N.D. ²	-
		0.000	N.D. ²	-
		0.010	0.0068	65.0
		0.010	0.0072	69.0
		0.020	0.0182	91.0
		0.020	0.0186	93.0

² N.D.: non-detected (less than 0.002 ppm)

Analytical Chemistry Branch
METHOD VALIDATION RESULTS
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Commodity	Chemical Added	PPM Added	PPM Found	Percent Recovery
Cattle Fat	DPX-KN128	0.000	N.D. ³	-
		0.000	N.D. ³	-
		0.010	0.0096	96.0
		0.010	0.0096	96.0
		0.749	0.7296	97.4
		0.750	0.8461	113
	IN-JT333	0.000	N.D. ⁴	-
		0.000	N.D. ⁴	-
		0.010	0.0090	90.0
		0.010	0.0084	84.0
		0.749	0.6899	92.1
		0.750	0.7936	106

³ N.D.: non-detected (less than 0.003 ppm)

⁴ N.D.: non-detected (less than 0.003 ppm)

Analytical Chemistry Branch
METHOD VALIDATION RESULTS
 PP#8F04948

Commodity	Chemical Added	PPM Added	PPM Found	Percent Recovery
Milk Cream	DPX-KN128	0.000	N.D. ⁵	-
		0.000	N.D. ⁵	-
		0.010	0.0084	84.0
		0.010	0.0078	78.0
		0.752	0.7244	96.3
		0.749	0.6772	90.4
	IN-JT333	0.000	N.D. ⁶	-
		0.000	N.D. ⁶	-
		0.010	0.0065	65.0
		0.010	0.0062	62.0
		0.752	0.6983	92.9
		0.749	0.6450	86.1

⁵ N.D.: non-detected (less than 0.003 ppm)

⁶ N.D.: non-detected (less than 0.004 ppm)

EPA ADDENDUM

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ACB # B98-(47-50)
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Conditions and Substitutions used by ACB

1. ACB used a Hewlett-Packard 1100 LC system with a single column switching valve. The SB-Cyano LC column could therefore not be simultaneously backflushed during the analytical separation on the ODS column. Following the transfer of analytes from the cyano column to the ODS column, the Cyano column was equilibrated with 60:40 acetonitrile:30mM phosphate buffer, pH 3.0 before switching both columns on-line and eluting with 60:40. The columns were thermostatted at 35 ° C.

2. ACB pre-washed the Millex-HV final extract filters with 50:50 ACN 30mM KH₂PO₄ buffer, pH 3.0 prior to sample filtration. This was necessary to remove a contaminant from the filter that was producing an HPLC chromatographic interference close to the retention time of DPX-KN128/DPX-KN127.

3. ACB used a vacuum manifold during the Sep-Pak C18 SPE cartridge clean-up for milk cream in place of the disposable syringes suggested in the method.

4. The method suggests the use of average response factors to determine sample concentrations. ACB determined sample concentrations from a ratio of sample responses to the average of standard responses that bracketed the samples.

5. DPX-MP062 standard responses were found to be linear over the range of 5 - 150 ng injected. DPX-KN128 standard responses were found to be linear over the range of 5 - 100 ng injected. IN-JT333 standard responses were found to be linear over the range of 5 - 100 ng injected. Sample analyses were conducted in this range.

ACB's Recommended Changes to the Method

The following changes to the method were agreed to in a teleconference held between several staff members of ACB and DuPont.

1. On page 25, the 'Cream Extraction Procedure...' step 3 will be revised to eliminate the sentence "Let the sample sit for 15 minutes to allow the solvent to evaporate." This sentence is a writing error and should not be included in the method.

2. Page 26, step 25 should be revised to say that the acetonitrile should be evaporated using either a RapidVap, TurboVap or equivalent apparatus. The term

EPA ADDENDUM

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'dryness' should be removed because there are fats and oils which are liquid at the evaporation temperatures and will not evaporate so the sample can not actually go to 'dryness'.

3. The 'Sep-Pak(R) C18 SPE....' section on pages 27 and 28 will be revised to allow the use of a vacuum manifold as well as the syringe approach already written into the method.

FIGURE
1

DPX-MP062 Standard Curve and Regression Data.

B98-(47-50) DPX-MP062 TMV

Compound: DPX-MP062
Instrument: HPLC:DAD
Date: 10/01/98

Linearity on Zorbax ODS HPLC Column @ 35 C

ug/mL	pk area 1	pk area 2	pk area 3	ave pk area	calc pk area
0.005	2.328	2.270	2.263	2.287	
0.010	3.888	4.685	4.771	4.448	
0.020	9.376	9.284	9.205	9.288	
0.100	44.603	43.097	44.212	43.971	
0.150	66.591	66.339	66.928	66.619	
0.000					0.136
0.170					75.255

Regression Output:

Constant	0.13565197
Std Err of Y Est	0.3047799
R Squared	0.99991495
No. of Observations	5
Degrees of Freedom	3

X Coefficient(s)	441.87628129
Std Err of Coef.	2.352828879

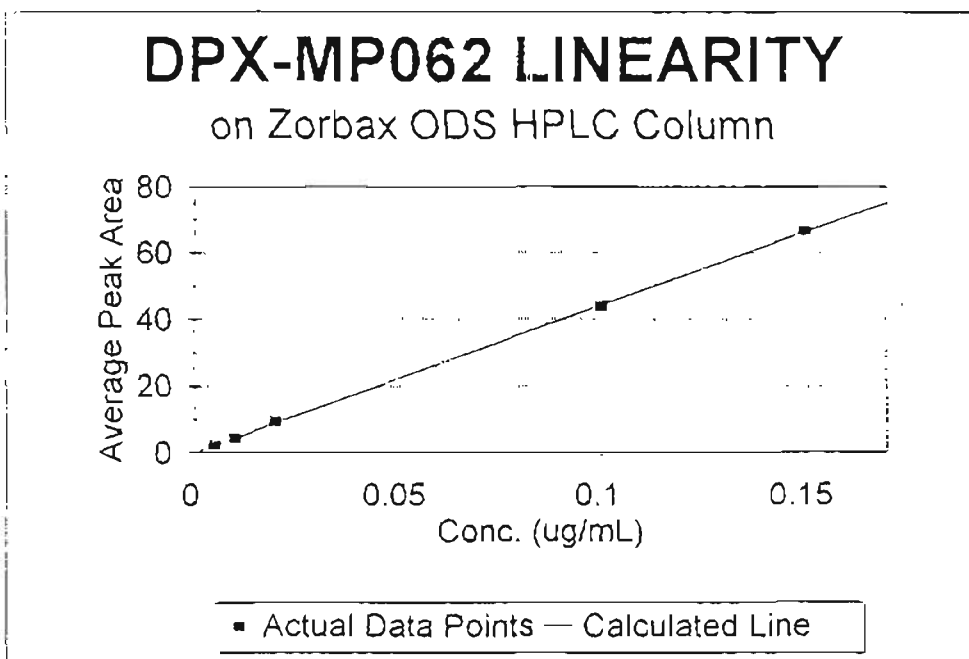


FIGURE # 3

IN-JT333 Standard Curve and Regression Data.

B98-(47-50) DPX-MP062 TMV

Compound: IN-JT333
Instrument: HPLC/DAD
Date: 04/27/99

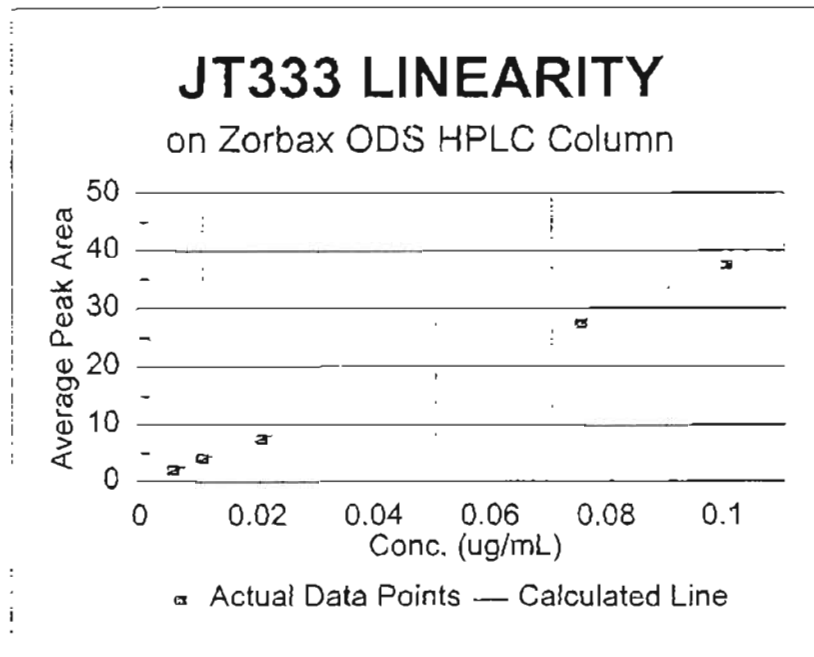
Linearity on Zorbax ODS HPLC Column @ 35 C

ug/mL	pk area 1	pk area 2	pk area 3	ave pk area	calc pk area
0.005	1.906	1.909	2.253	2.023	
0.010	3.968	4.119	3.859	3.982	
0.020	7.480	7.511	7.565	7.519	
0.075	28.020	27.245	27.053	27.439	
0.100	37.546	37.766	37.054	37.455	
0.000					0.155
0.110					40.826

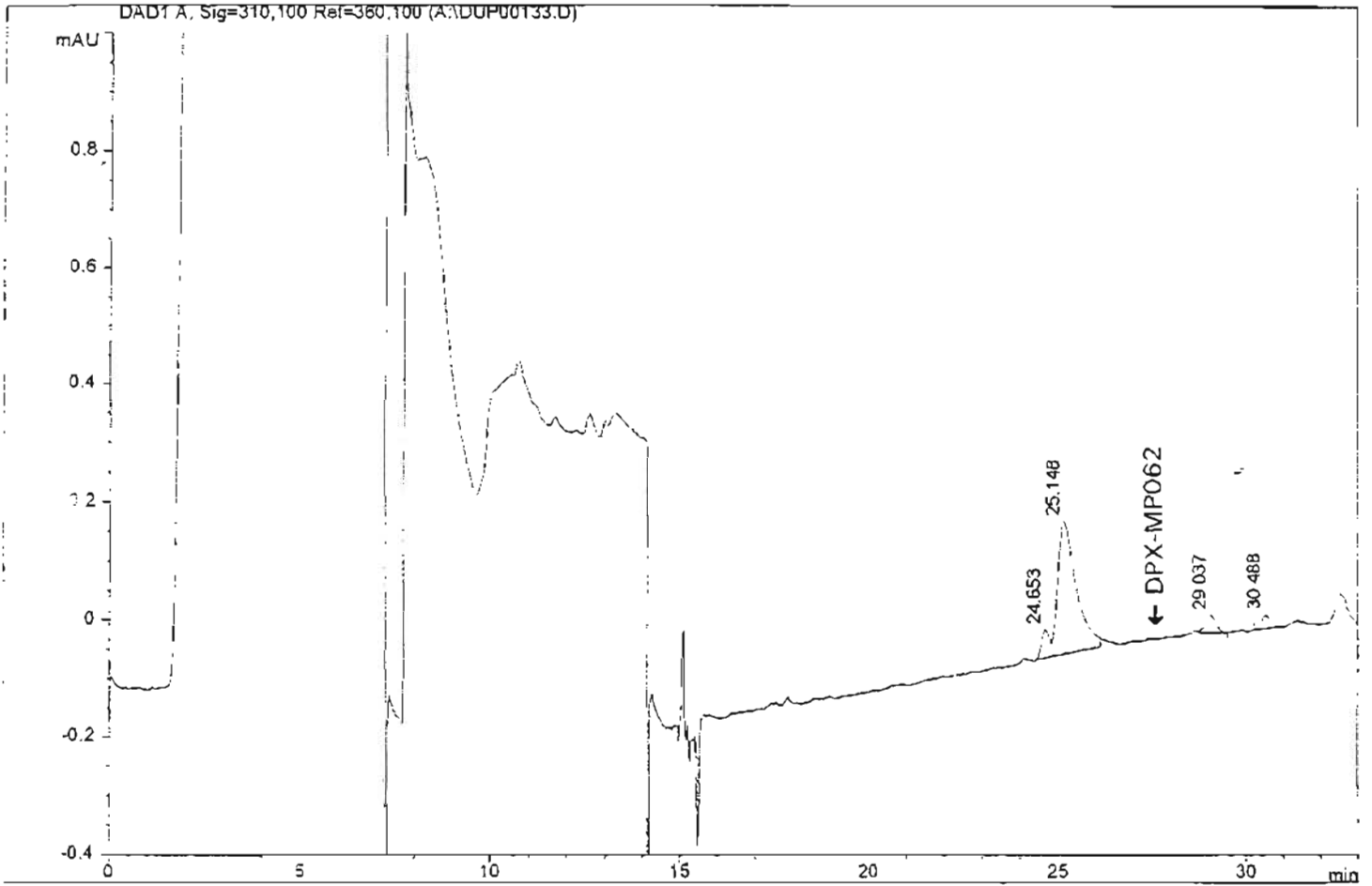
Regression Output:

Constant 0.15457844
Std Err of Y Est 0.32850894
R Squared 0.99967702
No. of Observations 5
Degrees of Freedom 3

X Coefficient(s) 369.7386
Std Err of Coef. 3.83703



CHROMATOGRAM Whole Milk Control - 5g milk /5 mL final volume.
2

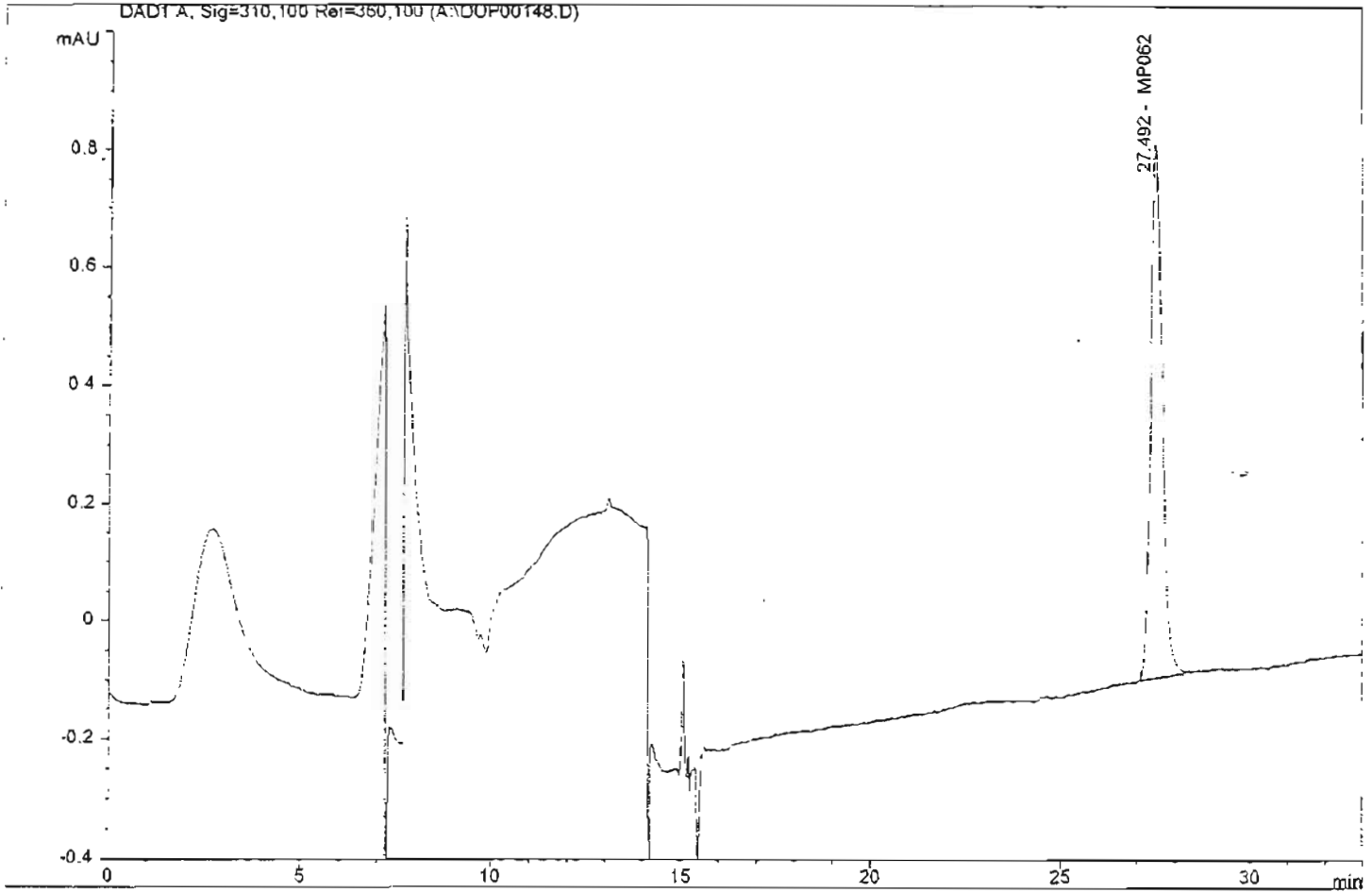


Injection Date : 12/2/1998 7:51:31 PM
 Sample Name : MC-4
 Sample Info. : 1000uL inj, 5.0 g whole milk, 5.0 mL F.V. 50:50 pH3 KH2
 PO4:ACN

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	24.653	PV	0.216	0.048	0.674	
2	25.148	VB	0.501	0.225	7.731	
3	0.000		0.000	0.000	0.000	MP062
4	29.037	BP	0.271	0.031	0.603	
5	30.488	PP	0.231	0.024	0.423	

CHROMATOGRAM # 4 Whole Milk, DPX-MP062 Standard - 0.10 ug/mL (0.10 ppm DPX-MP062 equivalent for a 5 mL final volume of a 5 g whole milk sample)

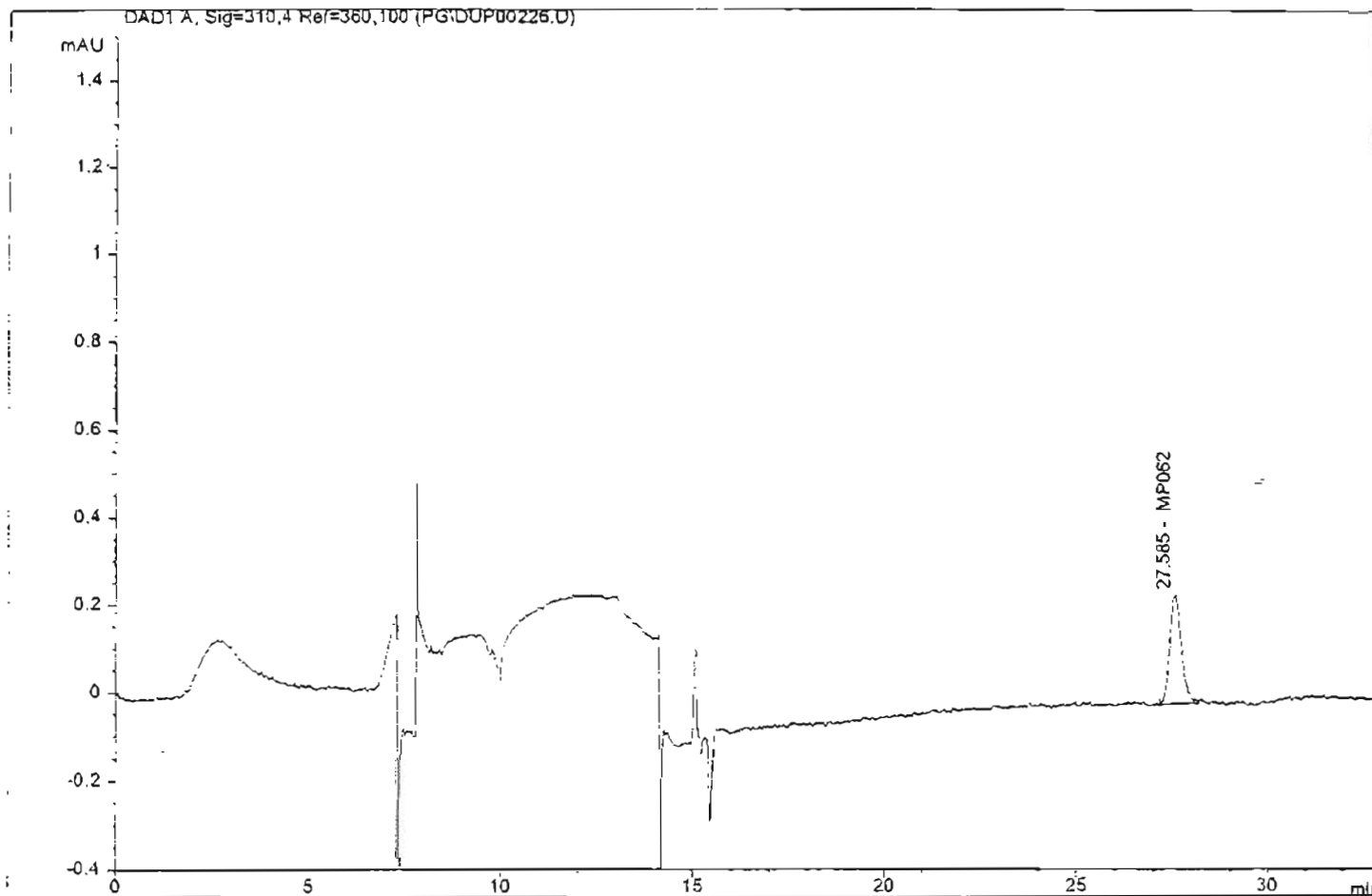


Injection Date : 12/3/1998 10:48:46 AM
 Sample Name : MP062 Std
 Sample Info. : 1000uL inj, 0.10 ug/mL in 50:50 pH3 KH2PO4:ACN

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	27.492	BB	0.320	0.908	18.962	MP062

CHROMATOGRAM # 6 Cattle Liver, DPX-MP062 Standard - 0.010 ug/mL (0.01 ppm DPX-MP062 equivalent for a 5 mL final volume of a 5 g liver sample.)

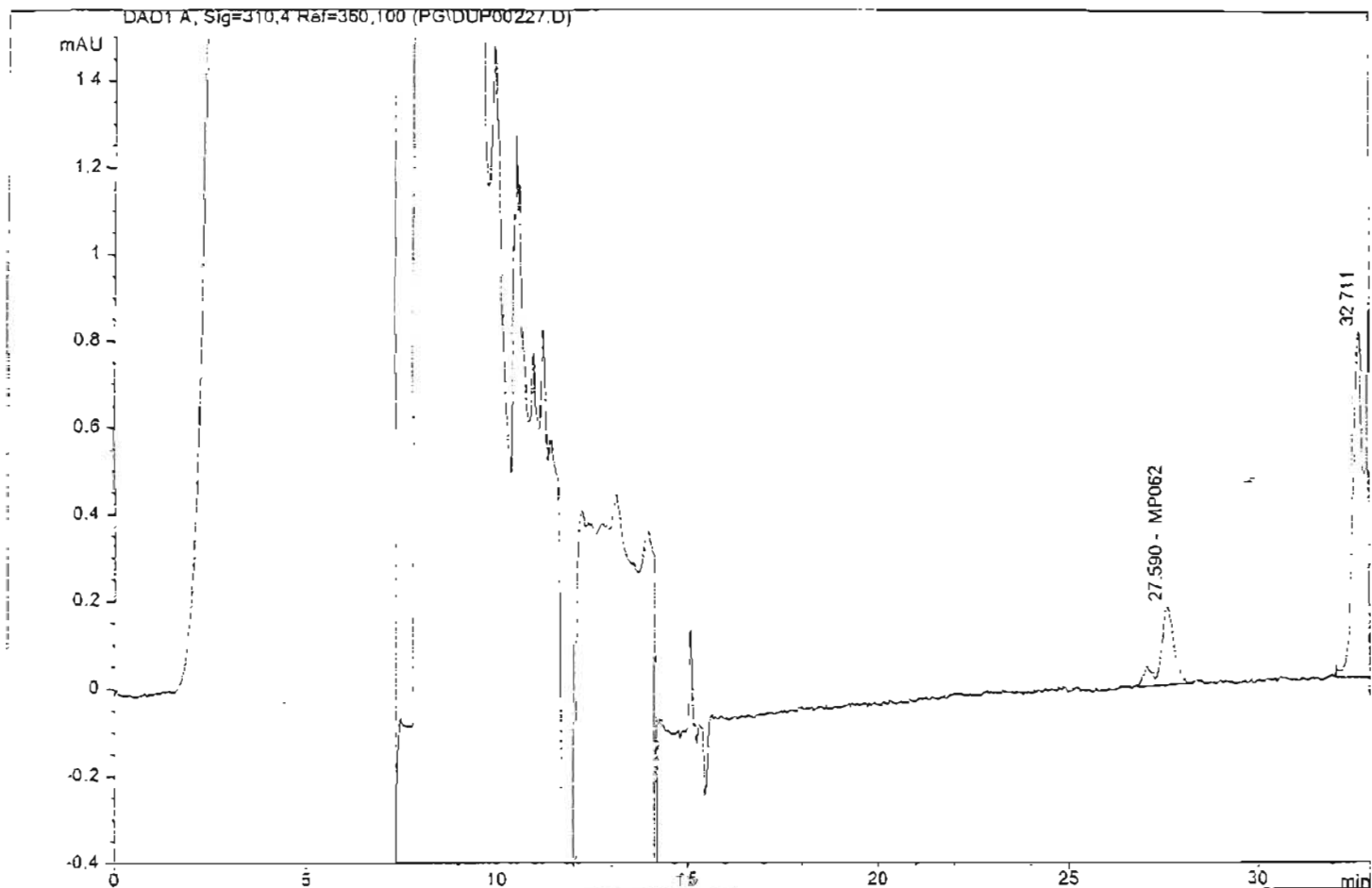


Injection Date : 1/21/1999 12:23:36 PM
 Sample Name : MP062 Std
 Sample Info. : 1000uL inj, 0.010 ug/mL in 50:50 pH3 KH2PO4:ACN

Signal 1 :DAD1 A, Sig=310,4 Ref=360,100

Peak #	RT (min)	Type	Width [min]	Height	Area	Name
1	27.585	PB	0.317	0.250	5.340	MP062

CHROMATOGRAM # 8 Cattle Liver, 0.01 ppm DPX-MP062 Fortification - 5g liver + 0.05 ug DPX-MP062 / 5 mL final volume.

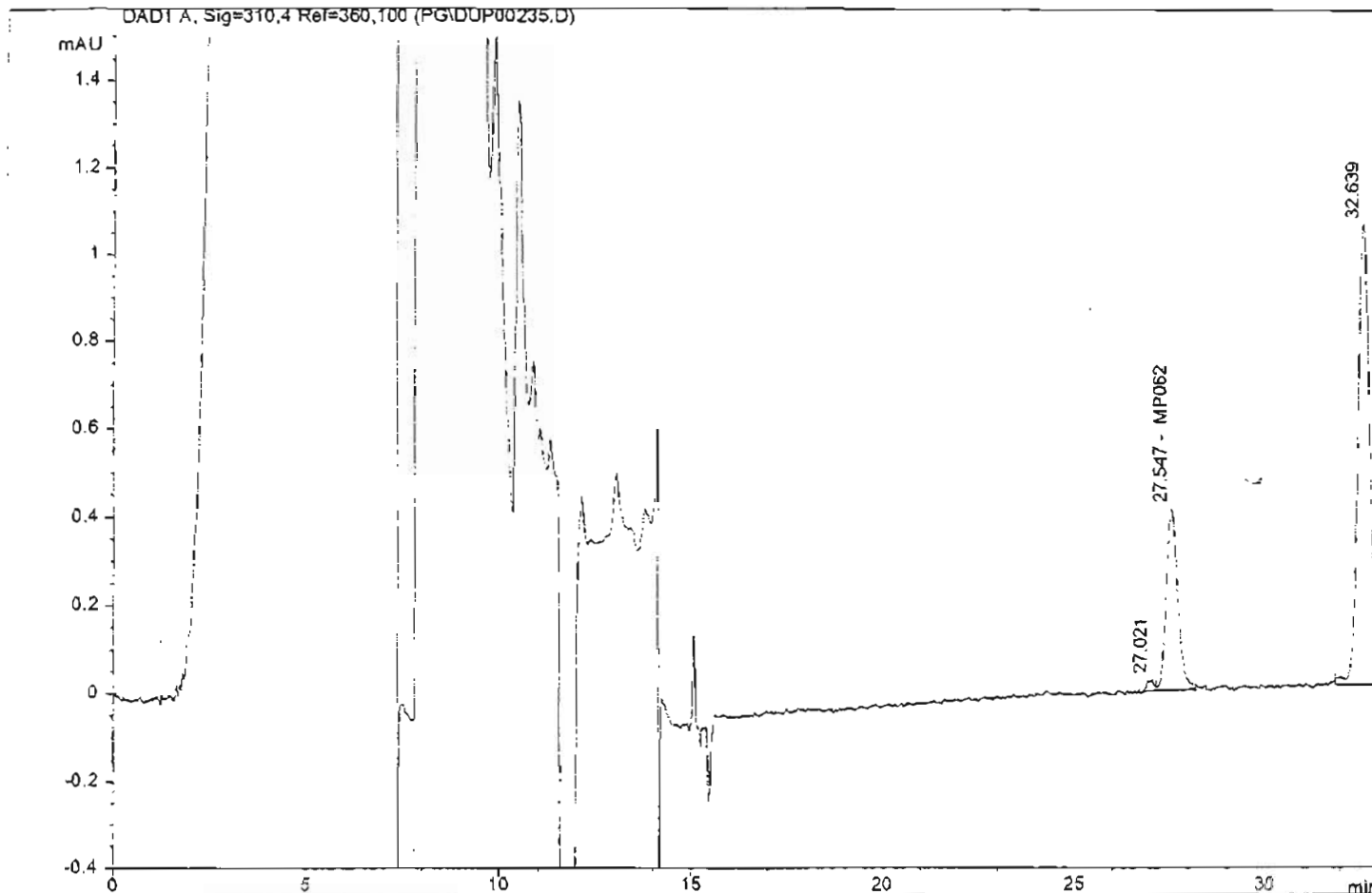


Injection Date : 1/21/1999 1:27:24 PM
 Sample Name : LL-1
 Sample Info. : 1000uL inj, 5.0 g cattle liver + 0.05 ug DPX-MP062 , 5.0 mL F.V. 50:50 pH3 KH2PO4:ACN, vial 2 of 3 filtered

Signal 1 :DAD1 A, Sig=310,4 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	27.051	BV	0.199	0.048	0.723	
2	27.590	VB	0.341	0.177	3.891	MP062
3	32.711	BBA	0.340	0.801	17.311	

CHROMATOGRAM Cattle Liver, 0.02 ppm DPX-MP062 Fortification - 5g liver + 0.10 ug DPX-MP062 /
 # 10 5 mL final volume.

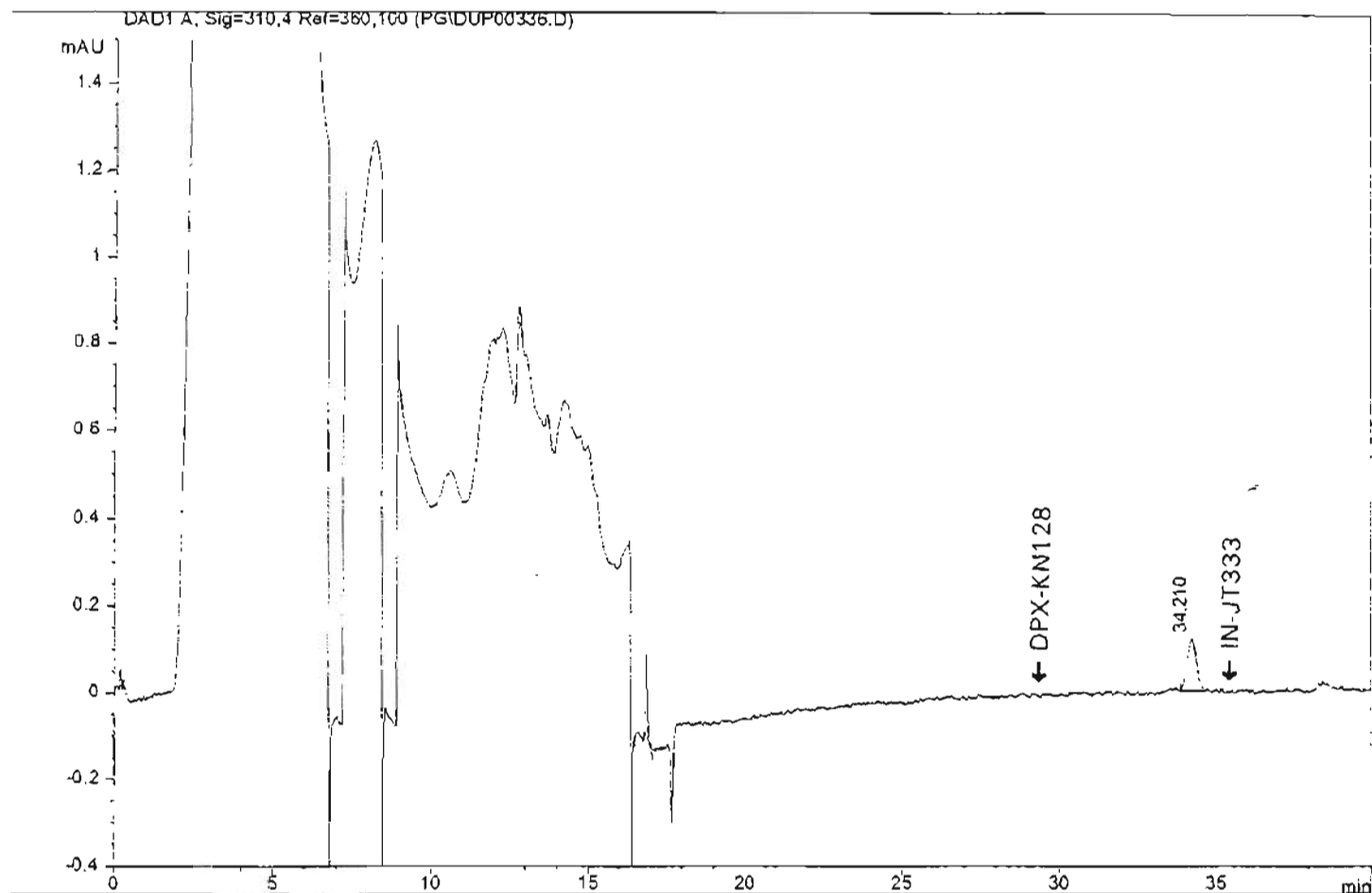


Injection Date : 1/21/1999 9:57:53 PM
 Sample Name : LH-1
 Sample Info. : 1000uL inj, 5.0 g cattle liver + 0.10 ug DPX-MP062 , 5.
 0 mL F.V. 50:50 pH3 KH2PO4:ACN, vial 2 of 3 filtered

Signal 1 :DAD1 A, Sig=310,4 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	27.021	BV	0.169	0.027	0.339	
2	27.547	VB	0.321	0.414	8.688	MP062
3	32.639	BBA	0.346	1.055	23.335	

CHROMATOGRAM Cattle Fat Control - 5g fat /5 mL final volume.
12

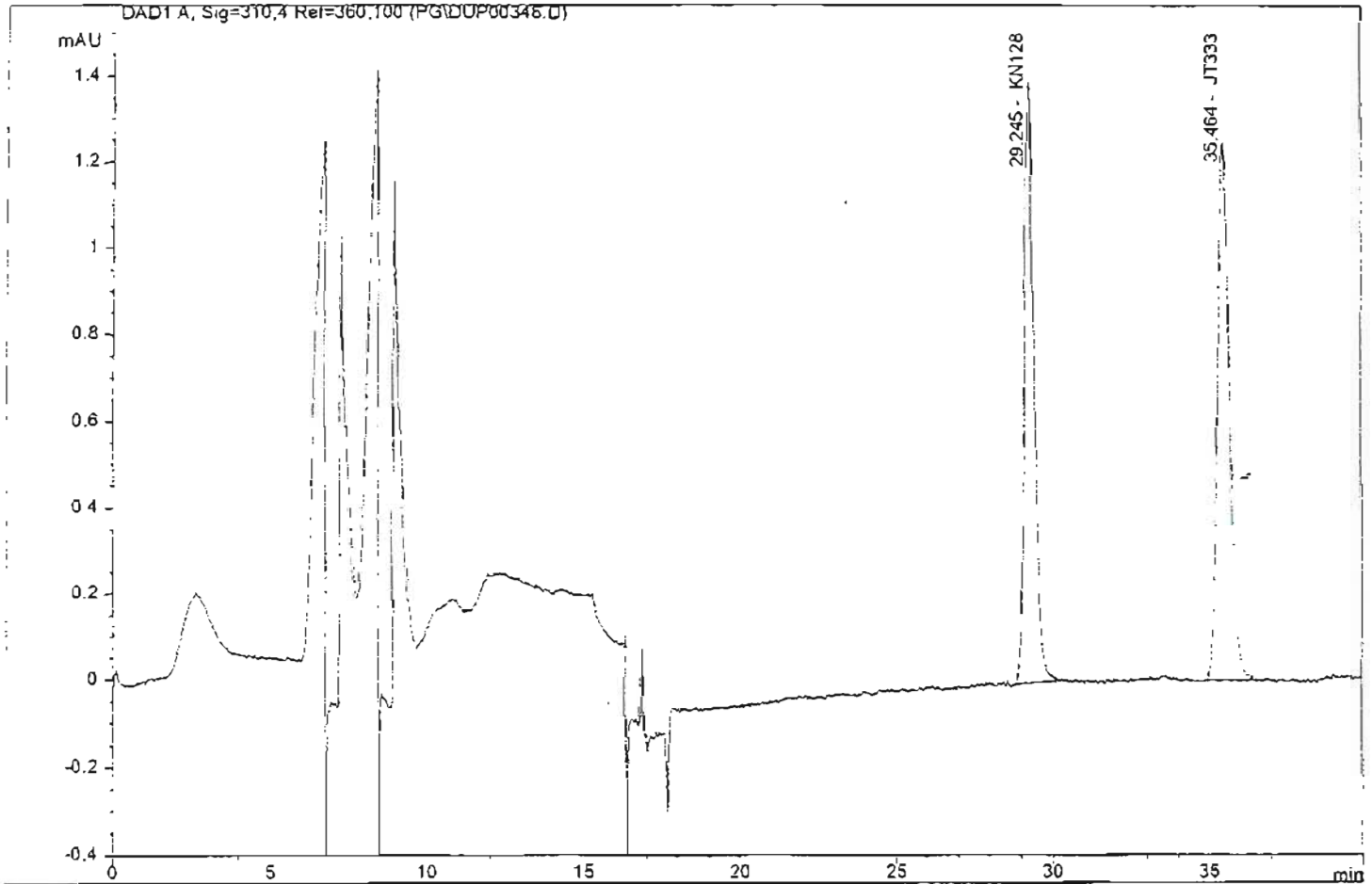


Injection Date : 5/5/1999 7:12:58 PM
 Sample Name : BFC-2(3)
 Sample Info. : 1000uL inj, 5.00g beef fat / 5.0mL final volume in 50:5
 0 pH3KH2PO4:ACN

Signal 1 :DAD1 A, Sig=310,4 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	0.000		0.000	0.000	0.000	KN128
2	34.210	BB	0.293	0.121	2.544	
3	0.000		0.000	0.000	0.000	JT333

CHROMATOGRAM # 14 Cattle Fat, DPX-KN128/IN-JT333 Mixed Standard - 0.075 ug/mL of each (0.75 ppm equivalents of each KN128 and JT333 for a 50 mL final volume of a 5 g fat sample.)

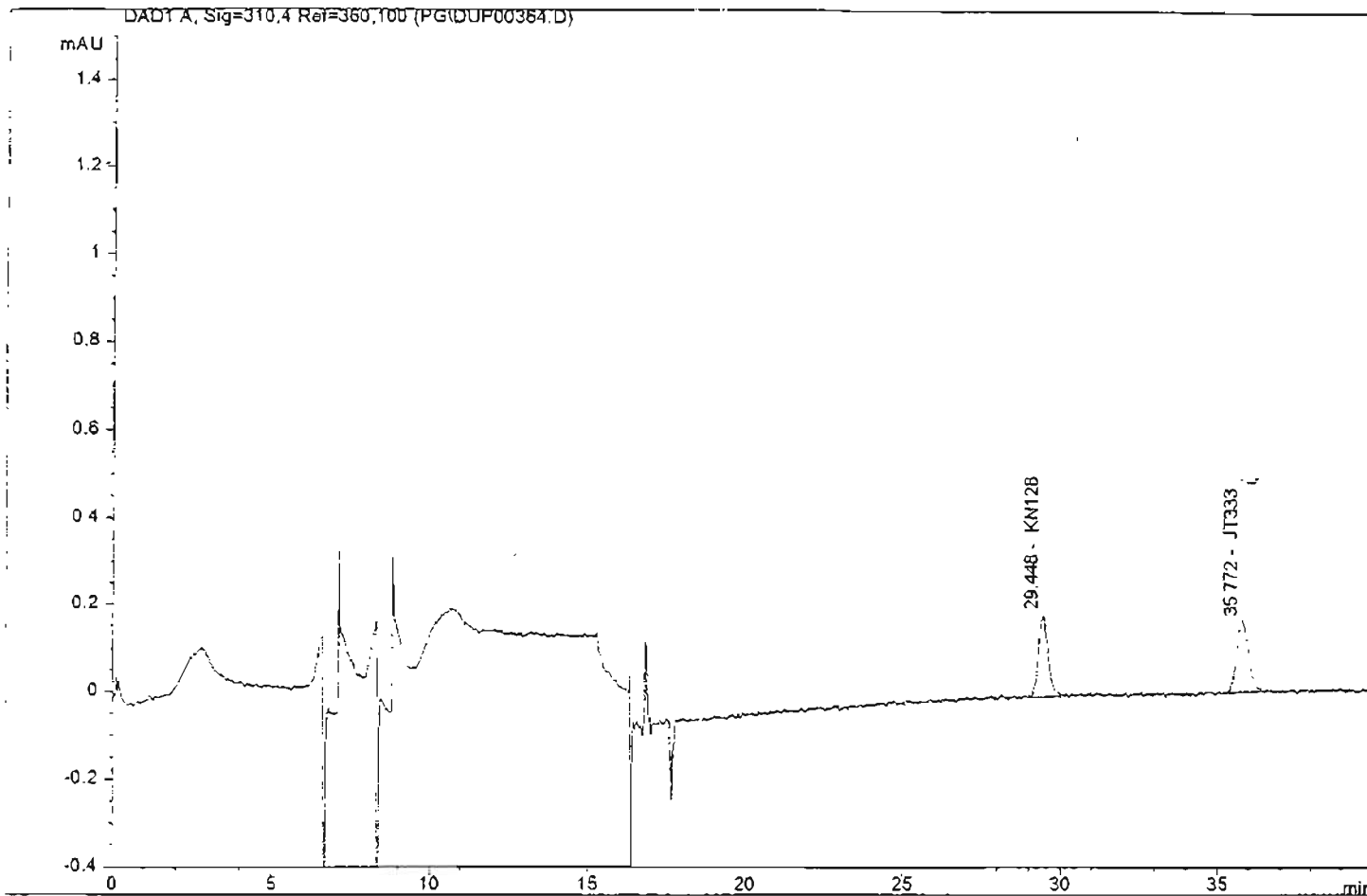


Injection Date : 5/6/1999 7:21:03 AM
 Sample Name : Mixed Std 4
 Sample Info. : 1000uL inj, 0.075 ug/mL each DPX-KN128 and IN-JT333 in 50:50 pH3 KH2PO4:ACN

Signal 1 :DAD1 A, Sig=310,4 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	29.245	BB	0.326	1.395	29.463	KN128
2	35.464	BB	0.414	1.248	33.537	JT333

CHROMATOGRAM # 16 Milk Cream, DPX-KN128/IN-JT333 Mixed Standard - 0.010 ug/mL of each (C.01 ppm equivalents of each KN128 and JT333 for a 5 mL final volume of a 5 g milk cream sample.)



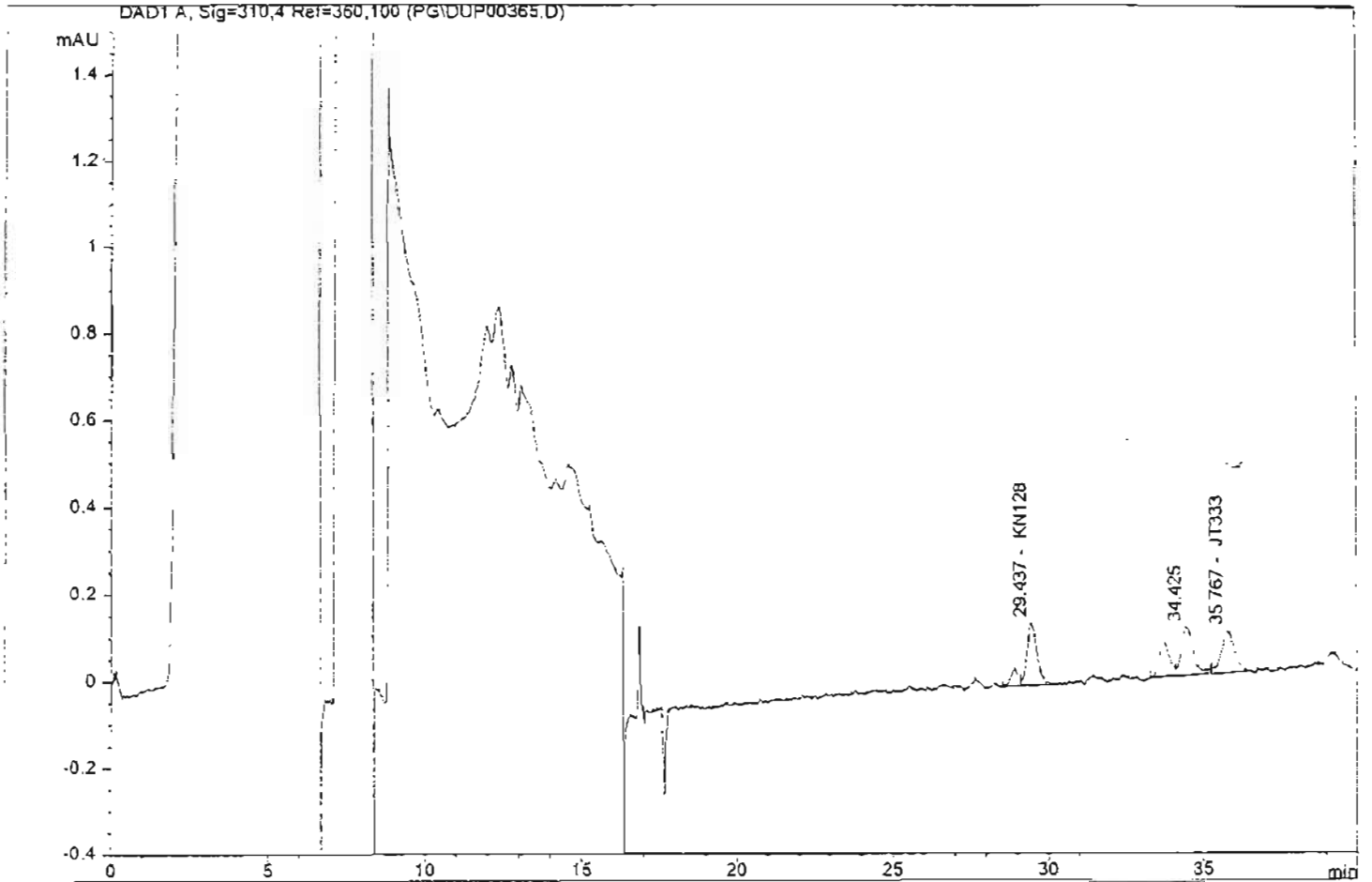
Injection Date : 5/13/1999 11:36:21 PM
 Sample Name : Mixed Std 2
 Sample Info. : 1000uL inj, 0.010 ug/mL each DPX-KN128 and IN-JT333 in
 50:50 pH3 KH2PO4:ACN

Signal 1 :DAD1 A, Sig=310,4 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	29.448	PB	0.317	0.187	3.984	KN128
2	35.772	BB	0.395	0.162	4.420	JT333

CHROMATOGRAM
18

Milk Cream, 0.01 ppm Fortification of each DPX-KN128 and IN-JT333 - 4.99g milk cream + 0.05 ug each KN128 and JT333 / 5 mL final volume.

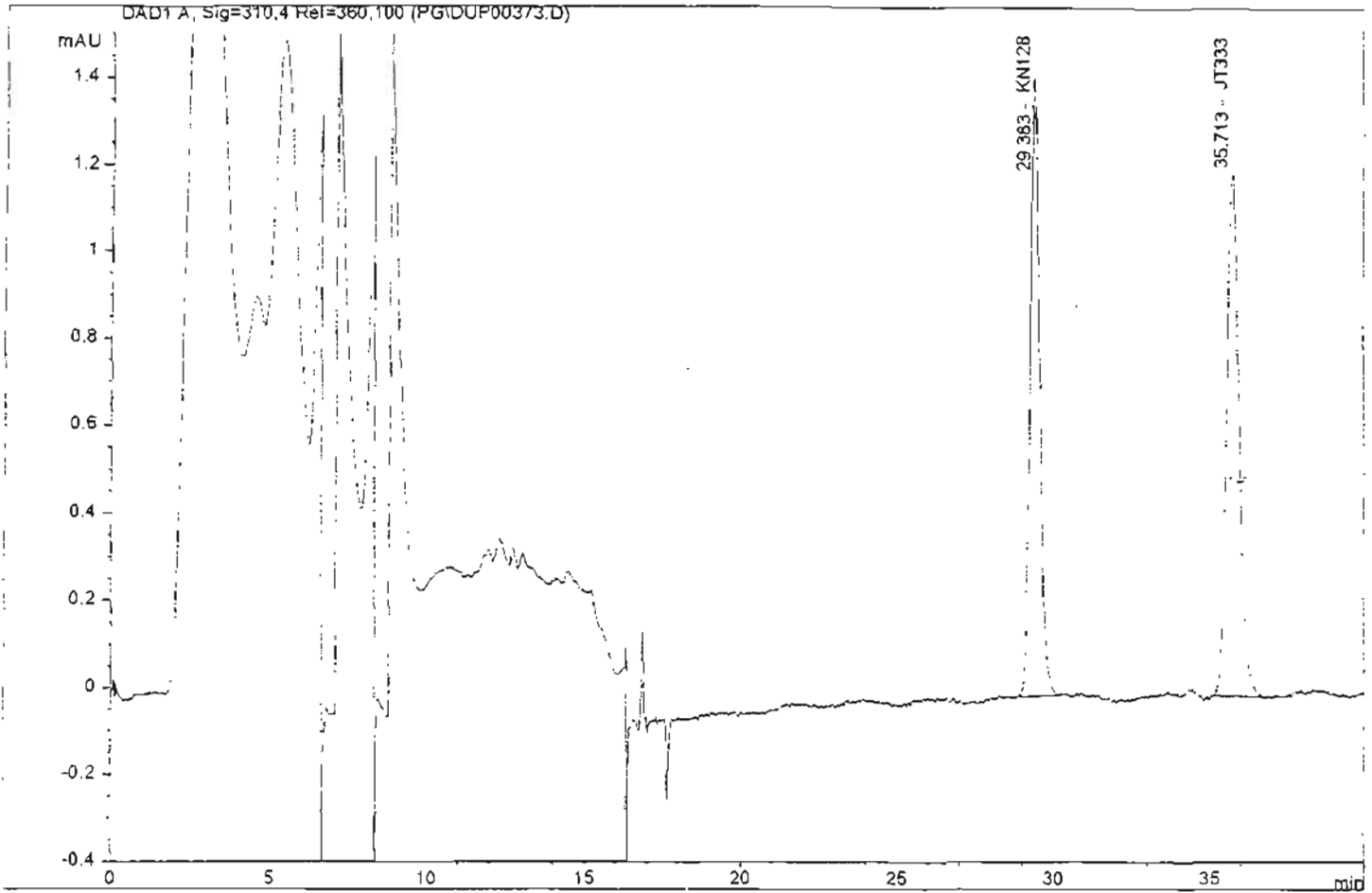


Injection Date : 5/14/1999 12:49:14 AM
 Sample Name : Cr-10-1(2)
 Sample Info. : 1000uL inj, 4.99g milk cream + 0.05ug each KN128 and JT333 / 5.0mL final volume in 50:50 pH3KH2PO4:ACN

Signal 1 :DAD1 A, Sig=310,4 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	28.894	PV	0.234	0.043	0.727	
2	29.437	VP	0.333	0.145	3.248	KN128
3	33.736	BV	0.340	0.078	1.881	
4	34.425	VB	0.369	0.111	2.914	
5	35.767	BB	0.363	0.097	2.838	JT333

CHROMATOGRAM Milk Cream, 0.752 ppm Fortification of each DPX-KN128 and IN-JT333 - 4.99g milk cream + 3.75 ug each KN128 and JT333 / 50 mL final volume.
 # 20



Injection Date : 5/14/1999 10:31:46 AM
 Sample Name : Cr-750-1(2)
 Sample Info. : 1000uL inj, 4.99g milk cream + 3.75ug each KN128 and JT
 333 / 50.0mL final volume in 50:50 pH3KH2PO4:ACN

Signal 1 :DAD1 A, Sig=310,4 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	29.383	BB	0.327	1.419	30.098	KN128
2	35.713	BP	0.426	1.203	33.212	JT333

ATTACHMENT 2



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

Analytical Chemistry Branch
Environmental Science Center
Fort Meade, MD 20755-5350

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

SEP 20 1999

MEMORANDUM

SUBJECT: PP# 8F04948. DPX-MP062 on Apples, Pome Fruit, Head and Stem Brassicas, Cotton, Leaf Lettuce, Head Lettuce, Fruiting Vegetables, and Sweet Corn. Tolerance Method Validation Request. B98/51-60, MRID# 444773-32. PRAT #289487

FROM: Diane M. Rains, Chemist *Diane M. Rains*
Douglas Swineford, Chemist *Douglas Swineford*
Analytical Chemistry Branch

THRU: Francis D. Griffith, Jr., Chief *Francis D. Griffith, Jr.*
Analytical Chemistry Branch

THRU: Donald A. Marlow, Laboratory Coordinator *Donald A. Marlow*
Biological and Economic Analysis Division (7503C)

TO: Karen Whitby, Branch Chief
Registration Action Branch I
Health Effects Division (7509C)
and
Arnold Lane, Branch Chief
Insecticide Branch
Registration Division (7505C)

INTRODUCTION

The Analytical Chemistry Branch (ACB) was requested by Registration Action Branch I (RAB 1) to conduct a Petition/Tolerance method validation (TMV) of the insecticide DPX-MP062 on apples, broccoli, cotton seed, cotton hulls, cotton oil, head lettuce, tomato, sweet corn, sweet corn forage, and sweet corn stover. DPX-KN128/DPX-KN127 (KN128/KN127) are the two isomers of the insecticide DPX-MP062, (R,S)-methyl 7-chloro-2,5-dihydro-2-[[[(methoxycarbonyl)[4-(trifluoromethoxy)phenyl]amino] carbonyl]indeno[1,2-e][1,3,4]

oxadiazine -4a(3H)-carboxylate. The E.I. DuPont de Nemours and Company, Inc. proposed tolerances on the requested commodities range from 0.02 to 25.0 ppm.

RAB1 requested that all samples be fortified in duplicate with DPX-MP062 at the following levels:

Commodity	Fortification Levels Requested by RAB1 (ppm)
apple	0.0, 0.2, 2.0
broccoli	0.0, 0.2, 10.0
cotton, seed	0.0, 0.2, 3.0
cotton, hulls	0.0, 0.2, 3.0
cotton, oil	0.0, 0.2, 3.0
head lettuce	0.0, 0.2, 7.0
tomato	0.0, 0.2, 0.7
sweet corn, kernel, + cob with husk removed	0.0, 0.2, 0.4
sweet corn, forage	0.0, 0.2, 20.0
sweet corn, stover	0.0, 0.2, 25.0

RECOMMENDATIONS

1. There has not been a successful TMV. ACB recommends that the method, as written, is unsuitable for the food tolerance enforcement of DPX-MP062 due to lack of clear instructions in the procedure.

2. The ACB recommends that the method not be forwarded to the U.S. Food and Drug Administration (FDA), or be made available for the State Laboratories as currently written.

3. The ACB recommends that as a condition of registration, DuPont be required to supply the appropriate amounts of DPX-MP062 to the National Pesticide Standards Repository at Fort Meade, Maryland as required by OPPTS Guidelines 830.1900 and 860.1650. The standard should be sent to the attention of Charles Stafford, EPA National Pesticide Standards Repository, Environmental Science Center, 701 Mapes Road, Fort Meade, MD 20755-5350.

4. The ACB recommends the changes suggested in the conclusion of this report be

considered when the method is revised.

5. The ACB defers judgement on the need for additional validation data until the revised method is reviewed.

CONCLUSIONS

1. The ACB concludes that the comments on silanization of glassware should be rewritten to note that problems with DPX-MP062 adhering to glassware in the absence of organic solvent have been noted. The ACB observed peaks were present in some of the controls which were found to be indicative of compound carryover from glassware. The method needs to be modified to state that glassware must be silanized.
2. The ACB also noted initially a problem with matching the petitioner's claimed sensitivity. Telephone conversation (telephone log available in project jacket folder) with both the petitioner and the ILV Laboratory revealed that both groups experienced this. The ACB was not able to match the sensitivity of the petitioner until sample extracts had been repeatedly injected onto the instrument. The ACB concludes that the method needs to be rewritten to include this information highlighted.

METHOD SUMMARY

The analytical method evaluated was titled: DuPont Report No. AMR 3493-95 Supplement No. 2, Revision No. 1, December 2, 1997 "Analytical Enforcement Procedure for the Analysis of DPX-KN127/DPX-128 in Crops and Related Process Fractions by GC-MSD" by Michael R. Gagnon, Richard A. Guinivan, and Paul J. Desmond. The independent laboratory validation study titled: DuPont Report No. AMR 4623-97, EN-CAS 97-0036, "Independent Laboratory Validation of a Proposed Tolerance Enforcement Analytical Method for the Determination of DPX-KN128/DPX-KN127 in Crops and Related Process Fractions by GC/MS" dated 2 October 1997, by Taffy T. Lyle, and John W. James was performed at EN-CAS Analytical Laboratories.

Sample aliquots of 5 grams were extracted with 20 milliliters of water and 150 milliliters of ethyl acetate. Samples were homogenized using a tissue homogenizer and then centrifuged at 2400 RPM for 15 minutes. A 30 milliliter aliquot of ethyl acetate (equivalent to 1 gram of sample) was taken and evaporated to dryness. The residue was dissolved in hexane and then passed through a silica SPE (solid phase extraction) column coupled with a carbon SPE cartridge. The collected fraction was then evaporated to dryness on an N-Evap. (nitrogen gas evaporator) Final extract was diluted in ethyl acetate and injected on a 5973 Hewlett-Packard capillary GC/MSD (gas chromatograph/mass selective detector). Instrumental parameters were set up as described in the method. Mass 527.2 was monitored. Quantitation was accomplished by standard bracketing technique, using electronic integration. A confirmatory method was reviewed as satisfactory which used the ratios of three ions.

The petitioner submitted copies of chromatograms showing standards, untreated controls, and fortified samples of lettuce, pear, apple wet pomace, and cabbage. The ILV (Independent Laboratory Validation) submitted copies of chromatograms showing standards, control and fortified tomato samples, and control and fortified spinach samples. Review of these chromatograms shows minimum interference at the retention time of DPX-MP062.

High recoveries beyond guideline recommendations (>120%) of various crops were observed. The method states that practical quantitation levels are dictated more by matrix enhancement of signal response than by signal to noise considerations. The method further notes that preparation of standards in matrix as a way around this problem. Finally, dilution of the final extract could also be used to reduce the matrix enhancement. EPA does not allow standards in matrix. Repeated injections of matrix onto the GC system was required to achieve the sensitivity needed to eliminate matrix interference problems by dilution of the final extract.

COMMENTS

1. The ACB calculated the LOQ's for the commodities tested (see table below). ACB concurs with the petitioner's assessment of 0.2 ppm as a practical quantitation limit for the method due to the problems with matrix enhancement.

Table 2

commodity	proposed tolerance (ppm)	ACB estimated LOD ¹ (ppm)	ACB calculated LOQ ² (ppm)
apple	2.0	0.06	0.18
tomato	0.70	0.03	0.09
lettuce (head)	7.0	0.01	0.03
broccoli	10.0	0.02	0.06

¹LOD estimated as one third calculated LOQ.

²LOQ calculated as ten times noise measured peak to peak at area near the retention time of DPX-MP062.

2. The ILV laboratory was EN-CAS Analytical Laboratory, Winston Salem, North Carolina. The ILV ran the petitioners method with two commodities. The ACB concludes there has been a successful ILV for the GC method.

3. Control samples were obtained from the local grocery store.

4. A set of six samples can be analyzed within 24 hours. This is with a gas chromatograph equipped with an autosampler.

5. Analytical standards used for this method validation were obtained from the petitioner (DuPont Ag Products). The unused portion of the standards will be retained by ACB in the National Pesticide Standard Repository.

ATTACHMENTS: 1.method validation results
2.additional data

cc: ACB File B98/51-60; Analyst-DRains.
7503C:ACB:ESC:DRains:2/9/99:(410)305-2908:edit:drains7/14/99.
RDI:QA Panel:2/3/99:TL:MLaw:7/12/99:BrCh:FGriffith:7/13/99.

Attachment 1

Analytical Chemistry Branch <u>Method Validation Results</u> PP# 8F4948				
Commodity	Chemical Added	ppm Added	ppm Found	% Recovery
apple	DPX-MP062	0.00	<0.1 ppm	
		0.00	<0.1 ppm	
		0.21	0.32 ppm	152*
		0.21	0.31 ppm	148*
		2.1	2.81 ppm	134*
		2.1	2.91 ppm	138*
broccoli	DPX-MP062	0.00	<0.15 ppm	
		0.00	<0.15 ppm	
		0.21	0.23 ppm	110
		0.21	0.20 ppm	95
		10.3	9.94 ppm	96
		10.3	9.76 ppm	95
head lettuce	DPX-MP062	0.00	<0.05 ppm	
		0.00	<0.05 ppm	
		0.21	0.28 ppm	133*
		0.21	0.24 ppm	114
		7.2	5.49 ppm	90
		7.2	7.43 ppm	103
tomato	DPX-MP062	0.00	<0.05 ppm	
		0.00	<0.05 ppm	
		0.21	0.24 ppm	114
		0.21	0.25 ppm	119
		0.72	1.00 ppm	139*
		0.72	1.12 ppm	156*

*Recovery values outside the acceptable Guideline limits for residue analytical methods for tolerance enforcement.

Attachment 2

Analytical Chemistry Branch <u>Results for Additional Crops analyzed</u>				
Commodity	Chemical Added	ppm Added	ppm Found	% Recovery
cotton, seed	DPX-MP062	0.00	<0.1 ppm	
		0.00	<0.1 ppm	
		0.21	0.20 ppm	95
		0.21	0.16 ppm	76
		3.1	3.01 ppm	97
		3.1	2.80 ppm	90
cotton, hulls	DPX-MP062	0.00	<0.1 ppm	
		0.00	<0.1 ppm	
		0.21	0.19 ppm	90
		0.21	0.25 ppm	119
		3.1	3.02 ppm	97
		3.1	2.97 ppm	96
cotton, oil	DPX-MP062	0.00	<0.1 ppm	
		0.00	<0.1 ppm	
		0.21	0.16 ppm	76
		0.21	0.17 ppm	81
		3.1	2.30 ppm	74
		3.1	2.14 ppm	69
sweet corn, kernel + cob with husk removed	DPX-MP062	0.00	<0.1 ppm	
		0.00	<0.1 ppm	
		0.21	0.26 ppm	124
		0.21	0.24 ppm	114
		0.41	0.46 ppm	112
		0.41	0.43 ppm	105
sweet corn, forage	DPX-MP062	0.00	0.22 ppm	
		0.00	<0.1 ppm	
		0.21	0.32 ppm	152
		0.21	0.24 ppm	114
		20.1	15.95 ppm	79
		20.1	18.04 ppm	90

Analytical Chemistry Branch				
Results for Additional Crops analyzed				
Commodity	Chemical Added	ppm Added	ppm Found	% Recovery
sweet corn, stover	DPX-MP062	0.00	0.22 ppm	
		0.00	0.30 ppm	
		0.21	0.36 ppm	171
		0.21	0.28 ppm	133
		24.8	23.97 ppm	97
		24.8	20.84 ppm	84

ATTACHMENT 3



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

Analytical Chemistry Branch
Environmental Science Center
701 Mapes Road
Fort Meade, MD 20755-5350

JUL 21 1988

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP#8F04948. DPX-MP062 on Apples, Pome Fruit, Head and Stem Brassicas, Cotton, Leaf Lettuce, Head Lettuce, Fruiting Vegetables, and Sweet Corn. Method Validation Request. MRID#444773-27, DP Barcode D245424

FROM: Elmer H. Hayes, Chemist *E.H. Hayes*
Analytical Chemistry Laboratory Branch

THRU: Francis D. Griffith, Jr., Chief
Analytical Chemistry Laboratory Branch *Francis D. Griffith, Jr.*

THRU: Donald A. Marlow, Laboratory Coordinator
Biological and Economic Analysis Division (7503C)

TO: Karen Whitby, Chief
Registration Action Branch-1
Health Effects Division (7509C)
And
Arnold Layne, Chief
Insectide Branch
Registration Division(7505C)

INTRODUCTION

The Analytical Chemistry Branch (ACB) was requested by the Registration Action Branch I to conduct a TMV of the insecticide DPX-MP062 (KN128/KN127) (S)-methyl 7-chloro-2,5 dihydro-2-[[methoxycarbonyl][4-(trifluoromethoxy)phenyl]amino]carbonyl =indeno[1,2-e][1,3,4]oxadiazine-4a(3H)-carboxylate in/on Apples, Broccoli, Cotton Seed, Cotton Hulls, Cotton Oil, Head Lettuce, Tomato, Corn Kernel(sweet), Corn Forage, and Corn Stover. The proposed tolerance for the residues of MP062 to be established was from a range of 0.02 -25 ppm. Specific limit of quantitation(LOQ) for each commodity is shown on the validation results page.

RECOMMENDATION

1. ACB finds this HPLC method suitable for tolerance enforcement of the insecticide DPX-MP062 in/on apples, broccoli, cotton seed, cotton hulls, cotton oil, head lettuce, tomatoes, sweet corn kernels, corn forage, and corn stover.
2. ACB recommends that as a condition of registration, DuPont, be required to supply the appropriate amounts of DPX-MP062 to the National Pesticide Standards Repository at Fort Meade, MD.
3. ACB recommends the method be made available to Federal and State pesticide enforcement laboratories and forwarded to FDA's Technical Editing Group for publication in a future revision to PAM II with a Roman numerical designation. There is no EPA Addendum for this method.

METHOD SUMMARY

The method submitted for validation for this project was titled, "Analytical Enforcement Method (HPLC/Column Switching/UV) for the Determination of Residues of DPX-KN128 and IN-KN127 in Crops", by Frank K. Klemens, James D. McVicker, John D. Radcliff, DuPont Report #AMR 2712-93, 08-AUG-1997, (MRID#444773-27)

In summary, ten grams of sample are weighed into 250 ml glass bottle and tissued for 3-15 minutes depending on matrix with 100 mls of hexane saturated acetonitrile and 50 mls of acetonitrile (ACN) saturated hexane. The samples are then centrifuged (except the oil sample) and the supernatant is transferred to a separatory funnel where the two layers are allowed to separate. The ACN layer is collected in a TurboVap/Rapid Vap flask and concentrated to at 1 ml. The hexane layer is discarded. The residue is reconstituted in 10 mls of ACN and applied to a SPE purification procedure that can vary from one to a three stacked array of preconditioned SPE's whose support could be Silica or Sax, depending on sample matrix. The collected effluent is then evaporated to dryness on a N-Vap where it is later reconstituted with a chosen volume of 50% ACN/buffer solution. The purified sample is then analyzed by the use of a multi-dimensional HPLC with UV absorbance detection at 310 nm. The isocratic HPLC method (one HPLC, one pump, and one switching valve) employs two columns in series. The first column

is used as a "clean-up" column from which MP062 is transferred to the second column. The transfer occurs during a ± 0.5 minute window in relation to the peak retention time (apex) of MP062. Once the analyte is transferred to the second column, the analytical separation occurs.

Independent Laboratory Validation (ILV) Data

ABC Laboratories, Inc., Columbia, Missouri and E.I. duPont de Nemours, Wilmington, Delaware performed the ILV on the method: "Independent Laboratory Validation of a Proposed Enforcement Analytical Method for the Determination of DPX-Kn128 and In-Kn127 in Crops by HPLC/Column Switching/UV". ABC laboratories validated the following: Fortification at 0.01 ppm in sweet corn kernels plus cob with husk removed (K+CWHR), cotton seed, and sweet corn forage and 0.05 ppm for cotton gin trash. Fortification were also made at the proposed tolerance level (3.0 ppm for cottonseed, 20.0 ppm for cotton gin trash, 0.02 ppm for K+CWHR, and 10 ppm for sweet corn forage). The ILV analysis was successfully validated for all matrices in the first trial. The ILV method was performed as written with no major modifications. ACB did not experience any major problems. ACB agrees there has been a successful ILV for this method.

CONCLUSIONS

- 1) The cotton, corn, fruit, and vegetable commodity HPLC method has been successfully validated by ACB. ACB concludes that this method meets the requirements for an enforcement method as defined in the Pesticide Test Guideline 860.1340.
- 2) Adequate ILV data were presented for commodities proposed by this method, but no ILV or petitioners data was presented for 0.01 ppm apples, broccoli, head lettuce and tomato. Where no ILV or petitioners data was presented ACB agreed with the petitioner to use the "Corn Forage" procedure in the method.
- 3) The method states the limit of quantitation (LOQ) of MP062 in cotton (seed, forage, hulls, meal, and refined oil) and sweet corn (kernel & cobs with husk removed [K+CWHR], and forage) is 0.01 ppm and the LOQ for cotton gin trash and sweet corn fodder (stover) is 0.05 ppm. ACB agrees with the registrant's LOQ. ACB estimated the approximate lower limits of detection (LOD) for MP062 is 0.0025 ppm for the commodities investigated. ACB concludes the method to be suitable to gather residue data from the LOQ of 0.01 and 0.05 ppm to at least the proposed tolerance level.

4a) The analytical standard was obtained from the petitioner, DuPont. The purity of DPX-MP062 was 97.1%. These standards were not available from the EPA Repository as of 5/8/98. The remaining unused portion of the standard will be retained by ACB in the National Pesticide Standards Repository at Fort Meade, MD.

4b) ACB concludes that as a condition of registration, DuPont, be required to supply the appropriate amounts of DPX-MP062 to the National Pesticide Standards Repository at Fort Meade, MD.

5) The matrices (cotton seeds, cotton oil, cotton hulls, corn kernels, corn forage, and corn stover) used for controls and samples were provided by DuPont Ag Products.

6) ACB obtained good recoveries for the initial trials on all commodities after running some repeat analysis for cotton seed oil, cotton seed hulls, corn forage, broccoli.

7) At least six matrix samples can be prepared for analysis by a single analyst in 4-6 hours and a 20-24 hour overnight instrument analysis time depending on the number of samples being analyzed at one time and no instrumental discrepancies occur during instrumental overnight runs.

9) This method was simple to follow, easy to investigate and analyze. There is no EPA Addendum for this HPLC method.

ATTACHMENTS: Detector Response
Method Validation Results

Analytical Chemistry Branch
 Method Validation Results
 PP#8F04948

Commodity	Chemical Added	PPM Added	PPM Found	% Recovery
apple	DPX-MP062	0.00	0.00458	
		0.00	0.00457	
		0.010	0.00866	88.6
		0.010	0.0119	119
		2.00	1.79	89.5
		2.00	1.78	88.0
broccoli	DPX-MP062	0.00	< 0.0025	
		0.00	< 0.0025	
		0.010	0.00658	65.8
		0.010	0.00673	67.3
		10.0	6.57	65.7
		10.0	6.75	67.5
cotton seed	DPX-MP062	0.00	< 0.0025	
		0.00	< 0.0025	
		0.010	0.0100	100
		0.010	0.0105	105
		3.00	3.13	104
		3.00	3.17	108
cotton hulls	DPX-MP062	0.00	< 0.0025	
		0.00	< 0.0025	
		0.010	0.00907	90.7
		0.010	0.00890	89.0
		3.00	3.23	108
		3.00	2.86	95.3

Analytical Chemistry Branch
 Method Validation Results
 PP#8F04948

Commodity	Chemical Added	PPM Added	PPM Found	% Recovery
cotton oil	DPX-MP062	0.00	0.00653	
		0.00	0.00278	
		0.010	0.00797	79.7
		0.010	0.00856	85.6
		3.00	2.91	97.0
		3.00	3.77	126
head lettuce	DPX-MP062	0.00	0.00	
		0.00	0.0108	
		0.010	0.00965	96.5
		0.010	0.00910	91.0
		7.00	6.36	90.9
		7.00	6.48	92.6
tomato	DPX-MP062	0.00	< 0.0025	
		0.00	< 0.0025	
		0.010	0.00912	91.2
		0.010	0.0717	71.7
		0.700	0.571	81.6
		0.700	0.518	74.0
sweet corn, kernel + cob with husk removed	DPX-MP062	0.00	< 0.0025	
		0.00	< 0.0025	
		0.010	0.0113	113
		0.010	0.0127	127
		0.020	0.0124	62.0
		0.020	0.0173	86.5

Analytical Chemistry Branch
 Method Validation Results
 PP#8F04948

Commodity	Chemical Added	PPM Added	PPM Found	% Recovery
sweet corn, forage	DPX-MP062	0.00	< 0.0025	
		0.00	< 0.0025	
		0.010	0.00893	89.3
		0.010	0.00842	84.2
		20.0	18.4	92.0
		20.0	19.1	95.5
sweet corn, stover	DPX-MP062	0.00	< 0.0025	
		0.00	< 0.0025	
		0.050	0.0406	81.2
		0.050	0.0445	89.0
		25.0	15.8	63.2
		25.0	17.0	68.0

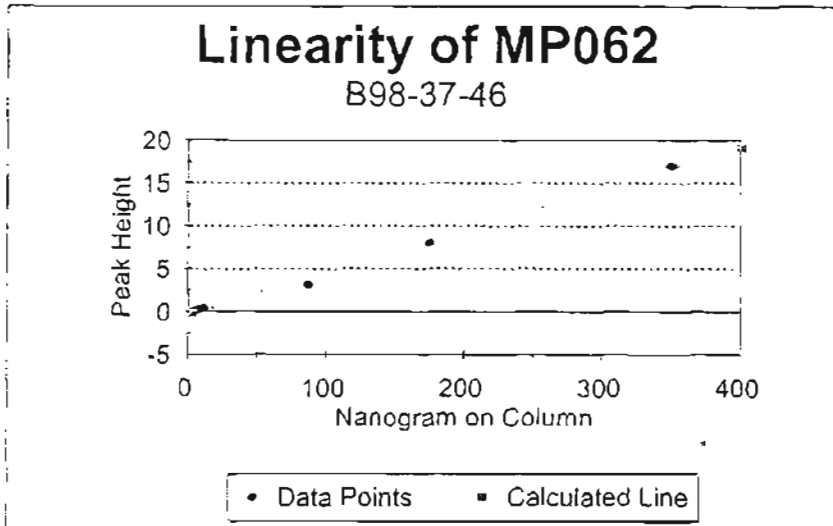
Linearity of MP062

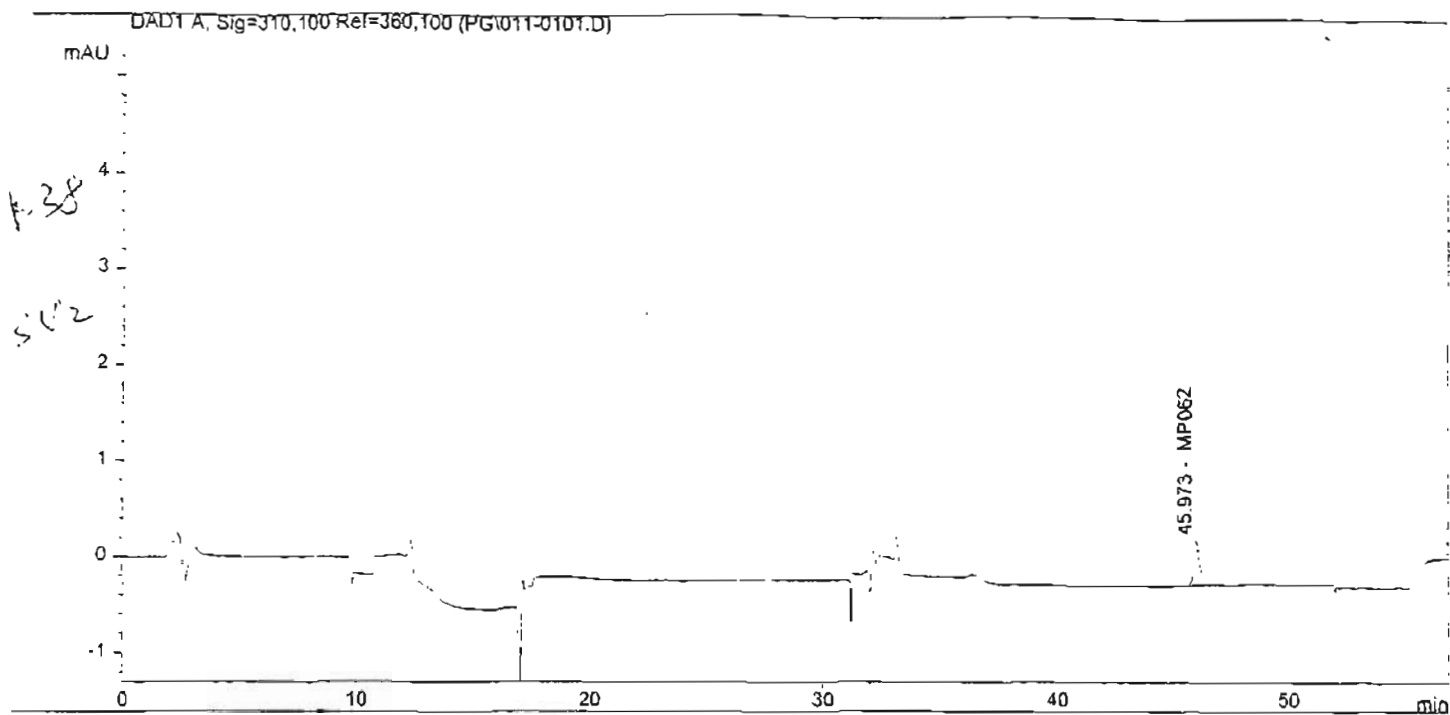
Date: 5/27/98
 Project #: B98-37-46
 Analyst: E.H. Hayes
 Instrument: HP 1100 HPLC
 Detector: UV-310 nm
 Column: Zorbax SB-CN/ ODS;(150mm x 4.6mm); 5u
 Solvent Phases: ACN/ BUFFER; changing compositions- 50/50-80-20, etc.
 Injection Volume: 100 ul

Standard Conc. (ug/mL)	Nanograms on Column	Peak Height 1	Peak Height 2	Average Peak Height	Calculated Regression Line
0.0525	5.25 ✓	0.224	0.227	0.226 ✓	
0.0700	7.00 ✓	0.299	0.302	0.301 ✓	
0.1050	10.50 ✓	0.457	0.458	0.458 ✓	
0.8750	87.50 ✓	3.746	2.591	3.169 ✓	
1.7500	175.00 ✓	8.116	8.034	8.075 ✓	
3.5000	350.00 ✓	na	17.075	17.075 ✓	
	2.500				-0.160 ✓
	400.000				19.235 ✓

Regression Output:	
Constant	-0.282203323 ✓
Std Err of Y Est	0.4885040926 ✓
R Squared	0.9957395875 ✓
No. of Observations	6
Degrees of Freedom	4
X Coefficient(s)	0.04879216047 ✓
Std Err of Coef.	0.00159577991 ✓

Calc.
CK by
JFH
9/10/98





=====
 B98-(37-46) DPX-MP062 TMV (Linearity Study)
 =====

Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name :MP062 STD(0.105)
 Sample Info. :STD. MP062--0.1050 ug/ml

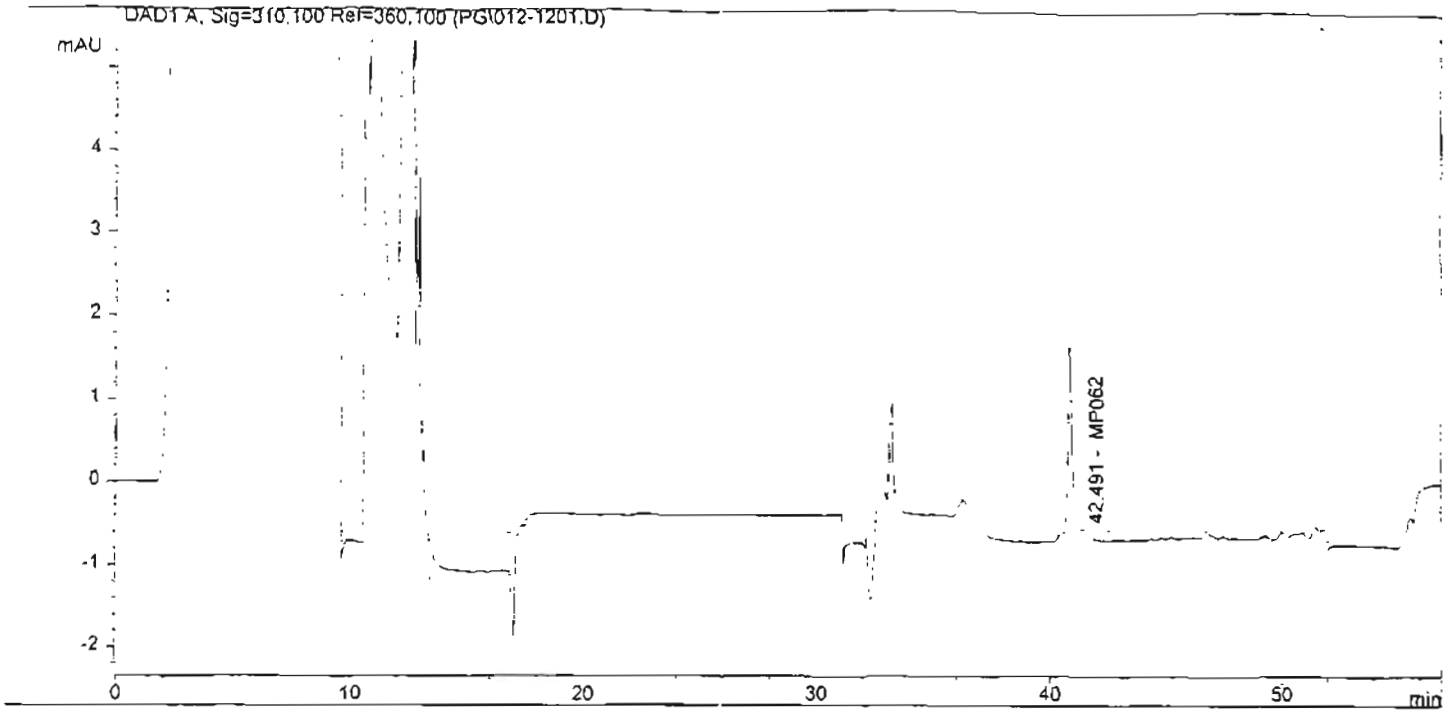
Injection Date :08/31/1998 13:05:43 PM
 Acq. Method :DUPONT3.M

Seq Line : 1
 Vial No. : 11
 Inj. number: 1
 Inj. Vol : 100 µl

Acq Operator :Elmer H. Hayes
 Analysis Method :C:\HPCHEM\1\METHODS\DUPONT3.M

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	45.973	BB	0.297	0.481	9.339	MP062



=====
 B98-(37-46) DPX-MP062 TMV (Linearity Study)
 =====

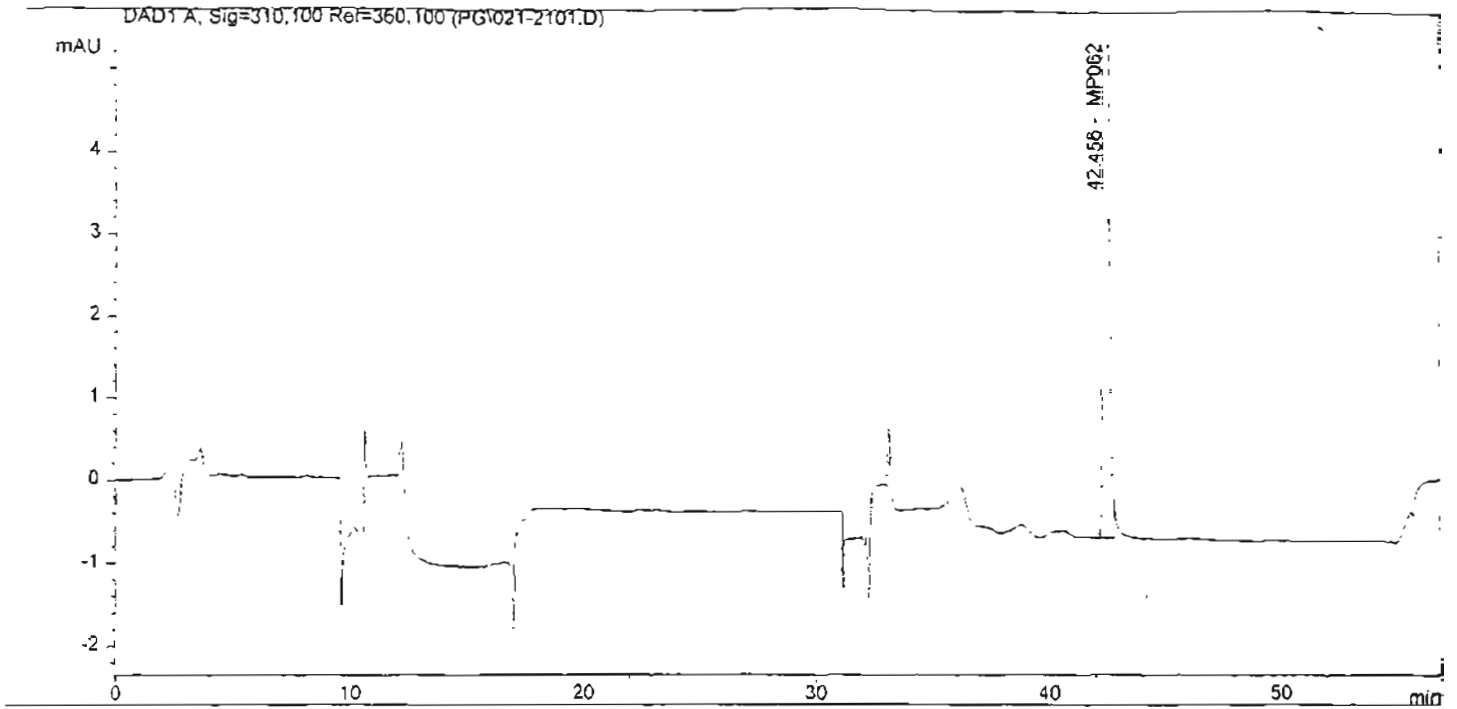
Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name : Apples-Cont-1
 Sample Info. : Control Apples # 1 sample--10g/1.5 ml final volume

Injection Date	: 08/26/1998 13:26:49 PM	Seq Line	: 12
Acq. Method	: DUPONT3.M	Vial No.	: 12
		Inj. number:	: 1
Acq Operator	: Elmer H. Hayes	Inj. Vol	: 100 µl
Analysis Method	: C:\HPCHEM\1\METHODS\DUPONT3.M		

Signal 1 : DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	42.491	PBA	0.188	0.144	1.794	MP062



=====
 B98-(37-46) DPX-MP062 TMV (Linearity Study)
 =====

Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

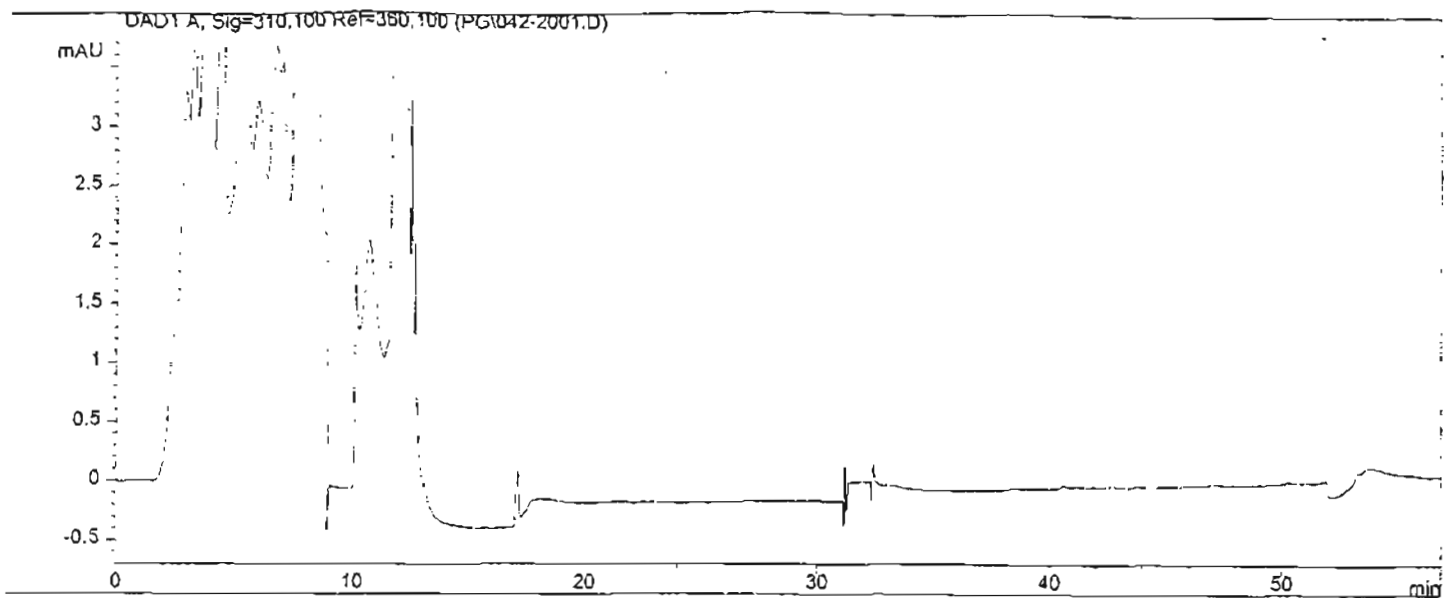
Sample Name :MP062 STD(2.63)
 Sample Info. :STD. MP062--2.63 ug/ml

Injection Date :08/27/1998 07:12:06 PM
 Acq. Method :DUPONT3.M
 Acq Operator :Elmer H. Hayes
 Analysis Method :C:\HPCHEM\1\METHODS\DUPONT3.M

Seq Line : 21
 Vial No. : 21
 Inj. number: 1
 Inj. Vol : 100 µl

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	42.456	BBA	0.203	18.528	242.948	MP062



B98-(37-46) DPX-MP062 TMV

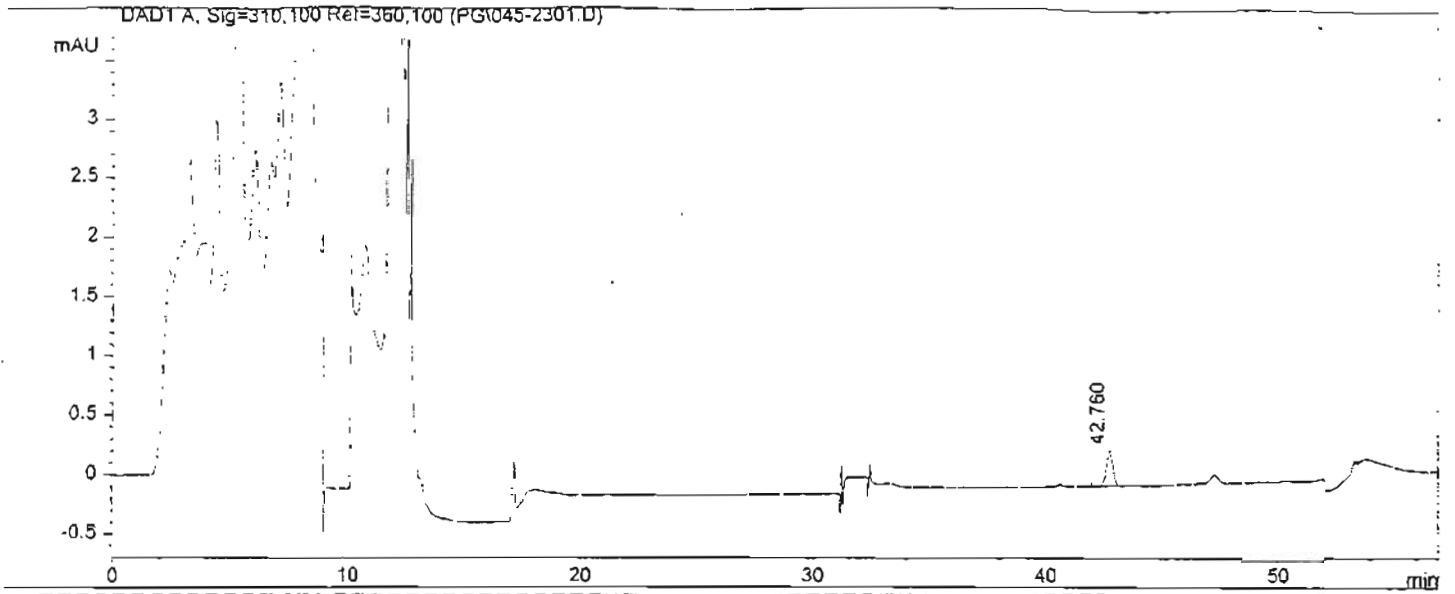
=====
 Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name :Cott-SControl -1
 Sample Info. :Control CottonSeed (ginned) # 1 sample--10g/1.5 ml fina
 1 volume

Injection Date :7/9/1998 15:42:04 PM Seq Line : 20
 Acq. Method :DUPONT3.M Vial No. : 42
 Inj. number: 1
 Acq Operator :E. Hayes/P. Golden Inj. Vol : 100 µl
 Analysis Method :C:\HPCHEM\1\METHODS\DUPONT3.M

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	0.000		0.000	0.000	0.000	MP062



B98-(37-46) DPX-MP062 TMV

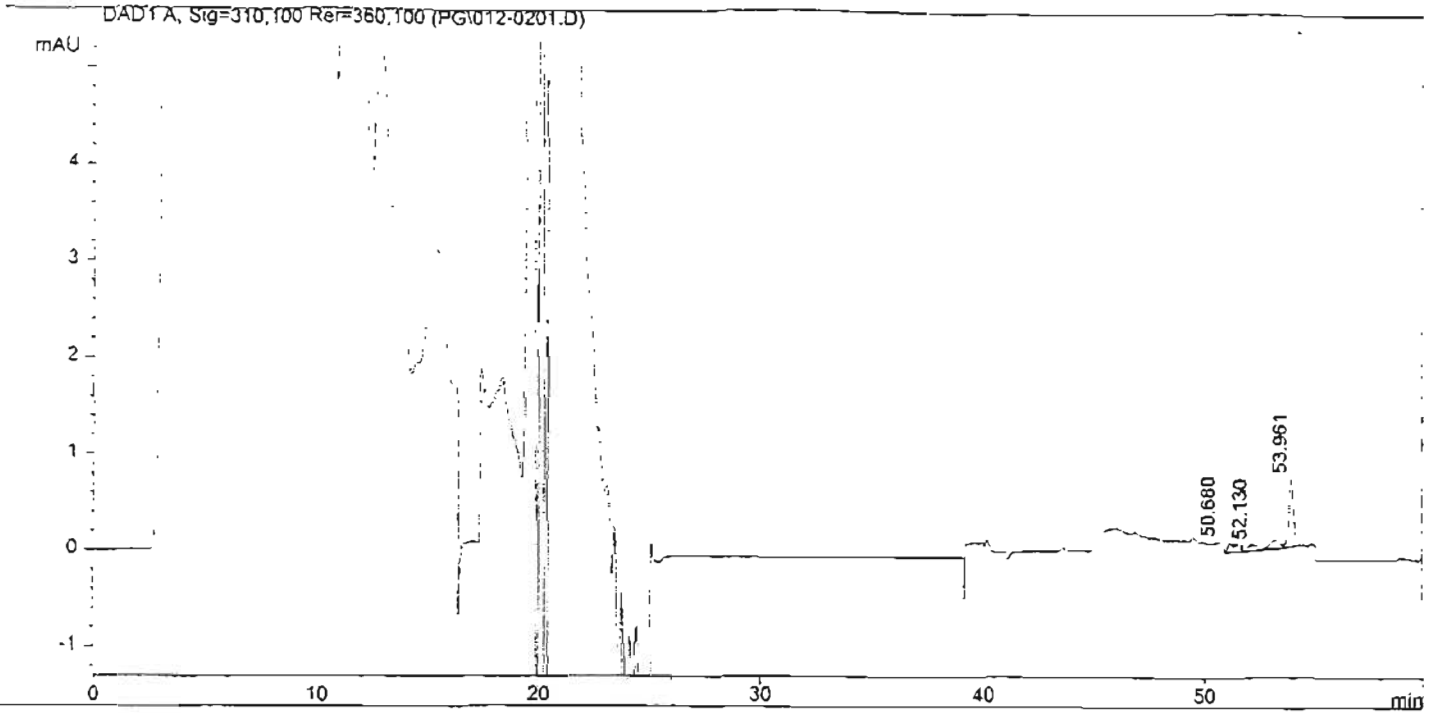
Analytical Enforcement Method (HPLC/Column Switching/UV) for the Determination of Residues of DPX-KN128 and IN-KN127 in Crops. ->

Sample Name :Cott-S 0.01-1
Sample Info. :CottonSeed (ginned), 0.01-1 ppm spike, 10g/1.5ml final volume

Injection Date :7/9/1998 21:37:25 PM Seq Line : 23
Acq. Method :DUPONT3.M Vial No. : 45
Inj. number: 1
Acq Operator :E. Hayes/P. Golden Inj. Vol : 100 µl
Analysis Method :C:\HPCHEM\1\METHODS\DUPONT4.M

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Table with 7 columns: Peak #, RT [min], Type, Width [min], Height, Area, Name. Row 1: 1, 42.760, BP, 0.297, 0.297, 5.889



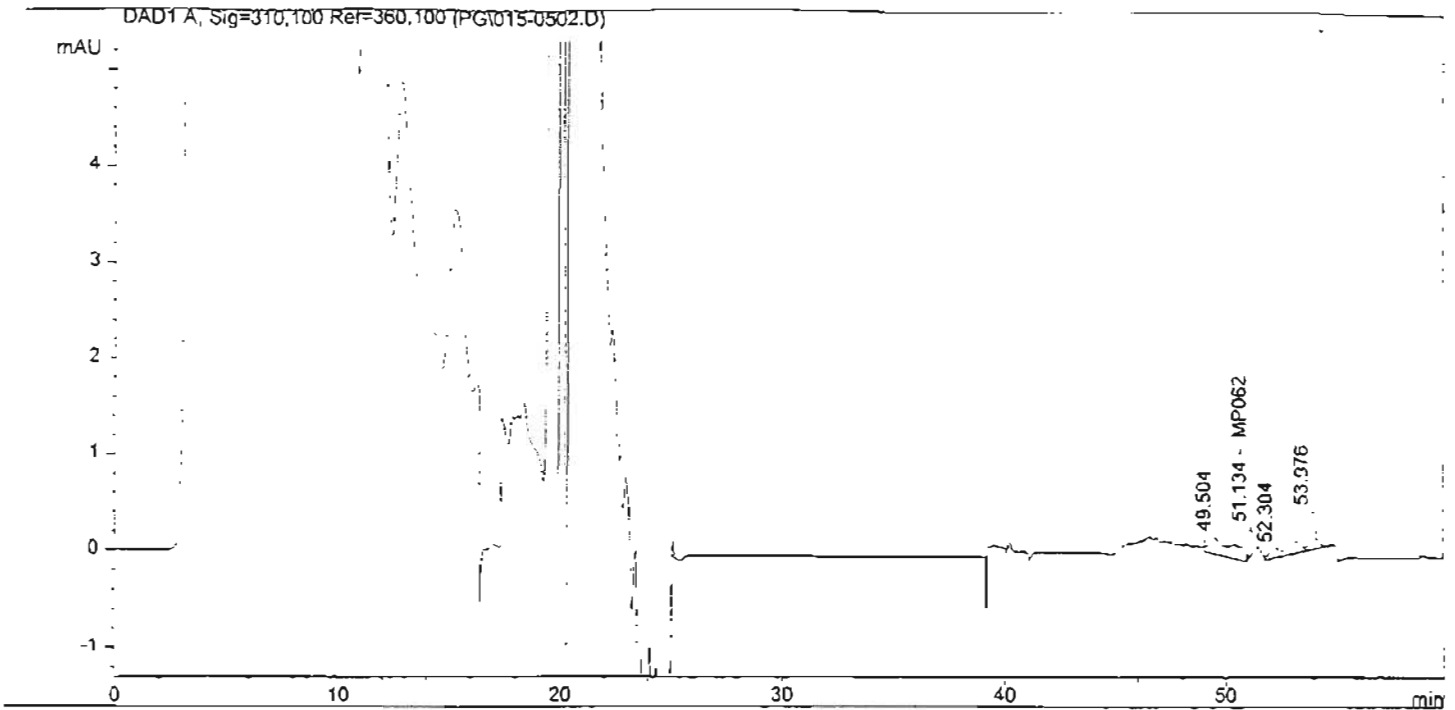
=====
 B98-(37-46) DPX-MP062 TMV (Linearity Study)
 =====

Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name : Broccoli-Cont-1
 Sample Info. : Control Broccoli # 1 sample--10g/1.5 ml final volume.
 Using 250 mm CN column.
 Injection Date : 09/08/1998 17:58:51 PM Seq Line : 2
 Acq. Method : DUPONT3.M Vial No. : 12
 Inj. number: 1
 Acq Operator : Elmer H. Hayes Inj. Vol : 100 µl
 Analysis Method : C:\HPCHEM\1\METHODS\DUPONT3.M

Signal 1 : DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	50.680	BP	0.267	0.067	1.380	
2	51.152	VV	0.206	0.100	1.384	MP062
3	51.439	VV	0.272	0.082	1.466	
4	52.130	VP	0.374	0.064	1.964	
5	53.175	VV	0.403	0.091	2.611	
6	53.961	VB	0.234	0.705	10.901	



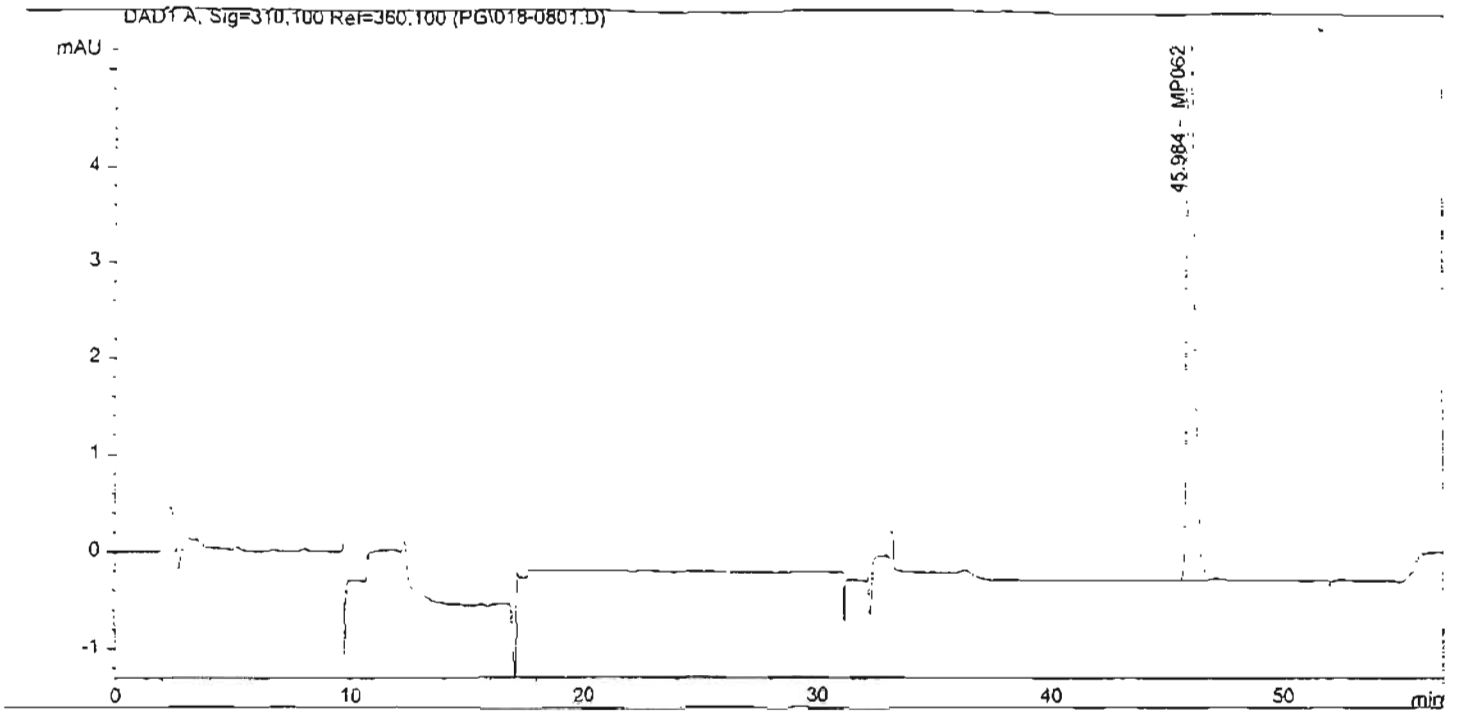
=====
 B98-(37-46) DPX-MP062 TMV (Linearity Study)
 =====

Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name : Broccoli-0.01-1
 Sample Info. : Broccoli, 0.01-1 ppm spike, 10g/1.5 ml final volume. U
 sing 250 mm CN column.
 Injection Date : 09/09/1998 01:14:01 PM Seq Line : 5
 Acq. Method : DUPONT3.M Vial No. : 15
 Inj. number: 2
 Acq Operator : Elmer H. Hayes Inj. Vol : 100 µl
 Analysis Method : C:\HPCHEM\1\METHODS\DUPONT3.M

Signal 1 : DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	49.504	BV	0.511	0.181	7.143	
2	50.326	VP	0.565	0.141	6.497	
3	51.134	MM	0.216	0.297 ✓	3.845	MP062
4	52.304	VV	0.430	0.092	3.265	
5	53.167	VV	0.477	0.119	4.592	
6	53.976	VB	0.266	0.399	7.267	



=====
B98-(37-46) DPX-MP062 TMV (Linearity Study)
=====

Analytical Enforcement Method (HPLC/Column Switching/UV) for the
Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

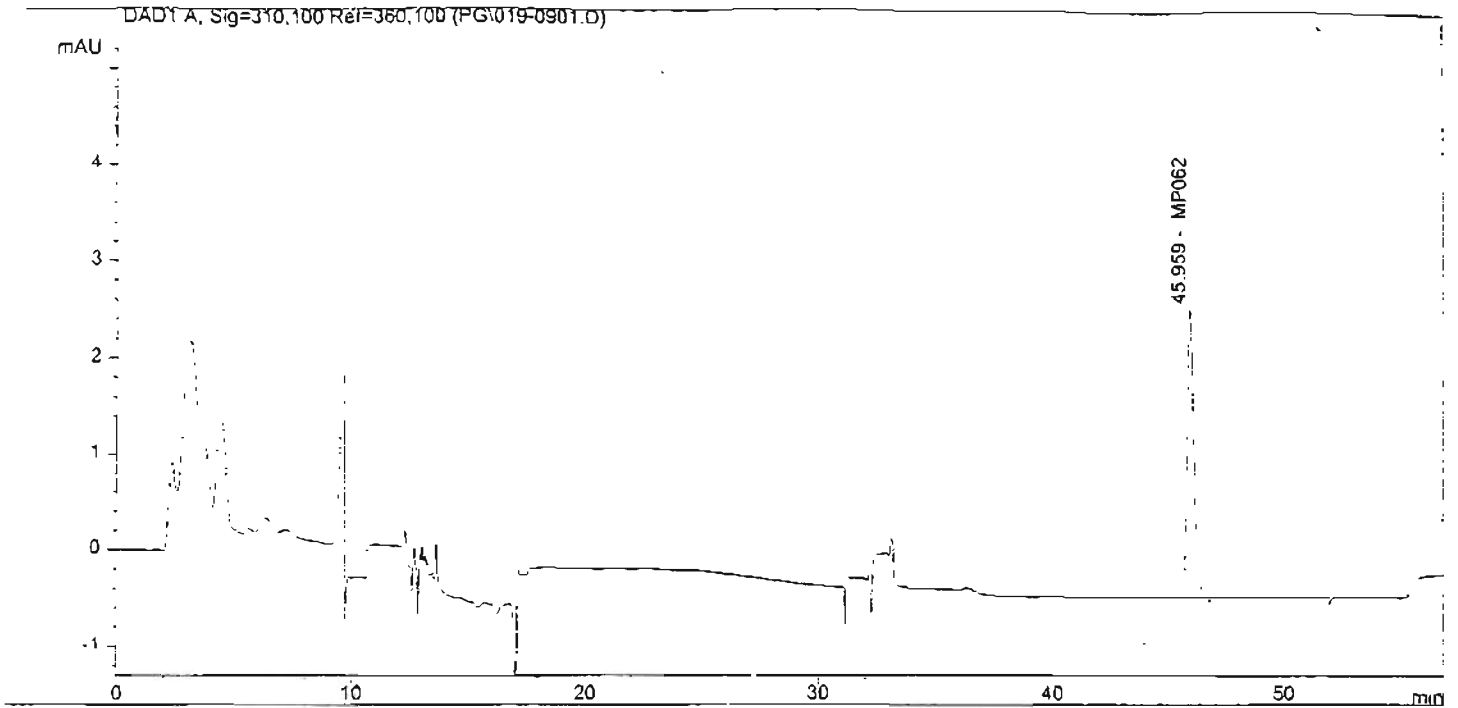
Sample Name :MP062 STD(1.75)
Sample Info. :STD. MP062--1.75 ug/ml

Injection Date :09/01/1998 14:07:06 PM
Acq. Method :DUPONT3.M
Acq Operator :Elmer H. Hayes
Analysis Method :C:\HPCHEM\1\METHODS\DUPONT3.M

Seq Line : 8
Vial No. : 18
Inj. number: 1
Inj. Vol : 100 µl

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	45.984	BB	0.296	7.987	155.021	MP062



=====
 B98-(37-46) DPX-MP062 TMV (Linearity Study)
 =====

Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name : Broccoli -10.0-1
 Sample Info. : Broccoli, 10 -1 ppm, 10g/100 ml final volume

Injection Date : 09/01/1998 16:05:29 PM Seq Line : 9
 Acq. Method : DUPONT3.M Vial No. : 19
 Acq Operator : Elmer H. Hayes Inj. number: 1
 Analysis Method : C:\HPCHEM\1\METHODS\DUPONT3.M Inj. Vol : 100 µl

Signal 1 : DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	45.959	BB	0.301	2.984	58.556	MP062

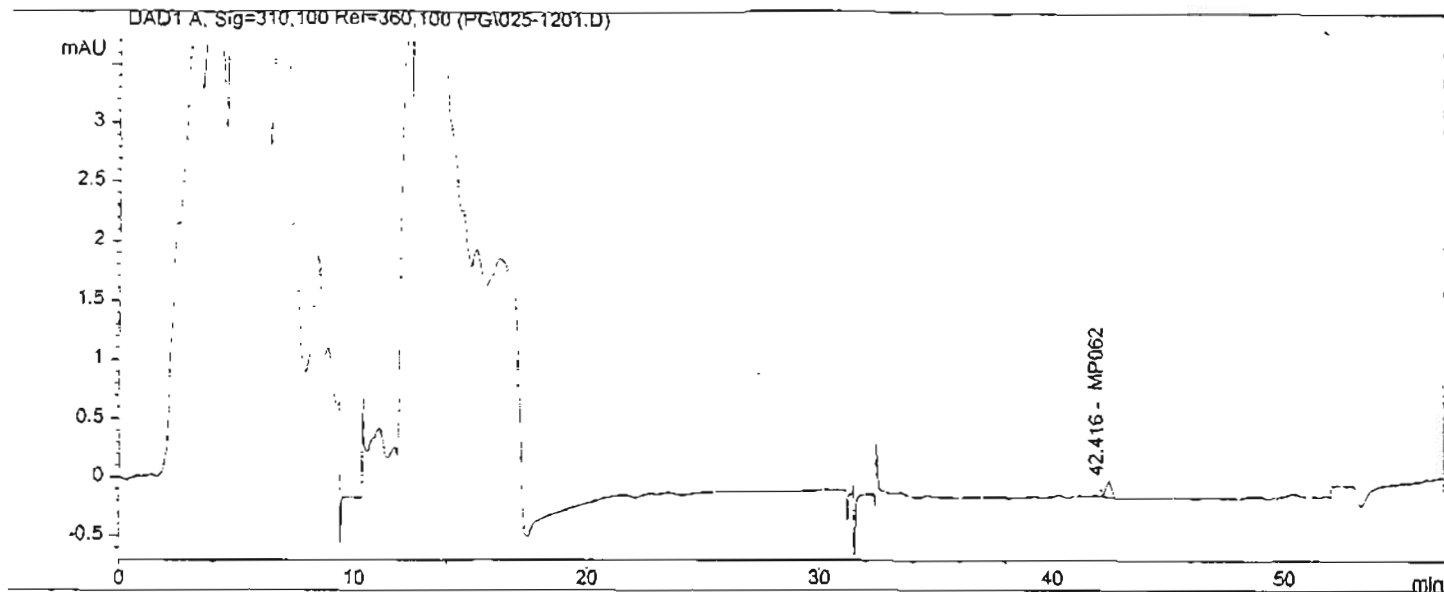


B98-(37-46) DPX-MP062 TMV (Linearity Study)

Analytical Enforcement Method (HPLC/Column Switching/UV) for the Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name :Cott-SControl -1
 Sample Info. :Control CottonSeed Oil # 1 sample--10g/1.5 ml final volume
 Injection Date :6/24/1998 16:12:38 PM Seq Line : 9
 Acq. Method :DUPONT3.M Vial No. : 22
 Inj. number: 1
 Acq Operator :E. Hayes/P. Golden Inj. Vol : 100 µl
 Analysis Method :C:\HPCHEM\1\METHODS\DUPONT3.M
 Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	0.000		0.000	0.000	0.000	MP062



=====
 B98-(37-46) DPX-MP062 TMV (Linearity Study)
 =====

Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

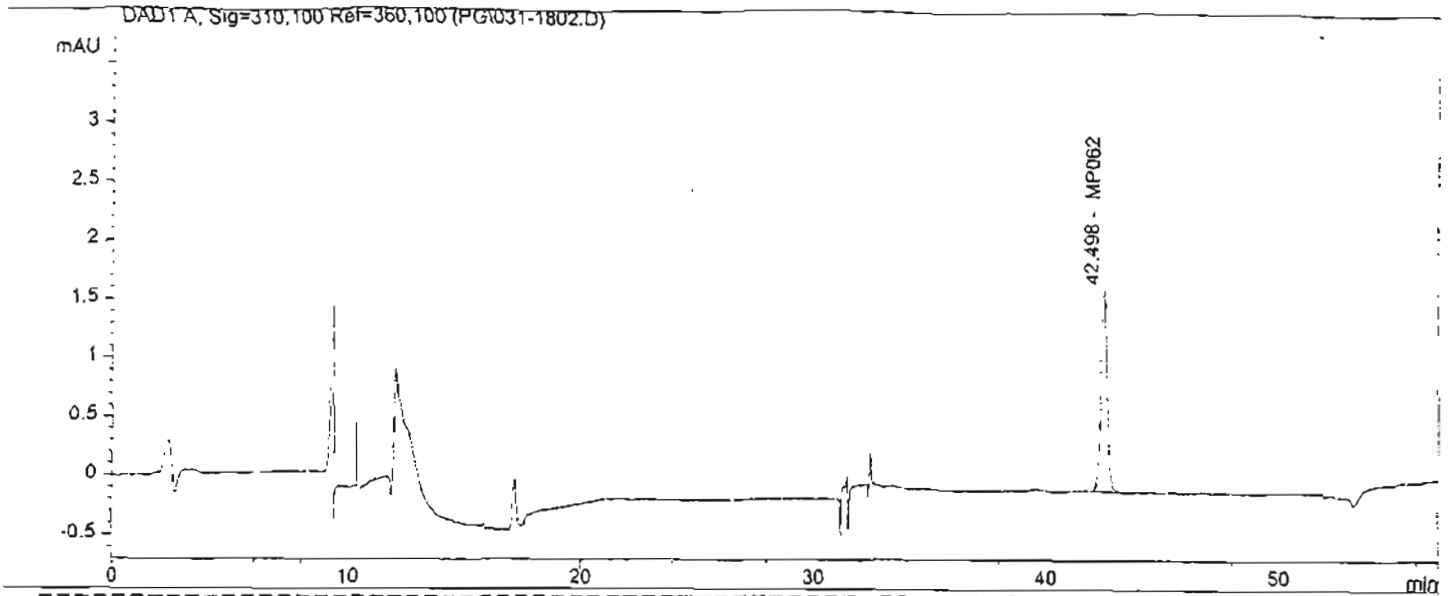
Sample Name :Cott-S 0.01-1
 Sample Info. :CottonSeed Oil, 0.01-1 ppm spike, 10g/1.5ml final volume

Injection Date :6/24/1998 22:07:46 PM Seq Line : 12
 Acq. Method :DUPONT3.M Vial No. : 25
 Acq Operator :E. Hayes/P. Golden Inj. number: 1
 Inj. Vol : 100 µl

Analysis Method :C:\HPCHEM\1\METHODS\DUPONT3.M

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	42.416	BP	0.285	0.140	2.605	MP062



B98-(37-46) DPX-MP062 TMV (Linearity Study)

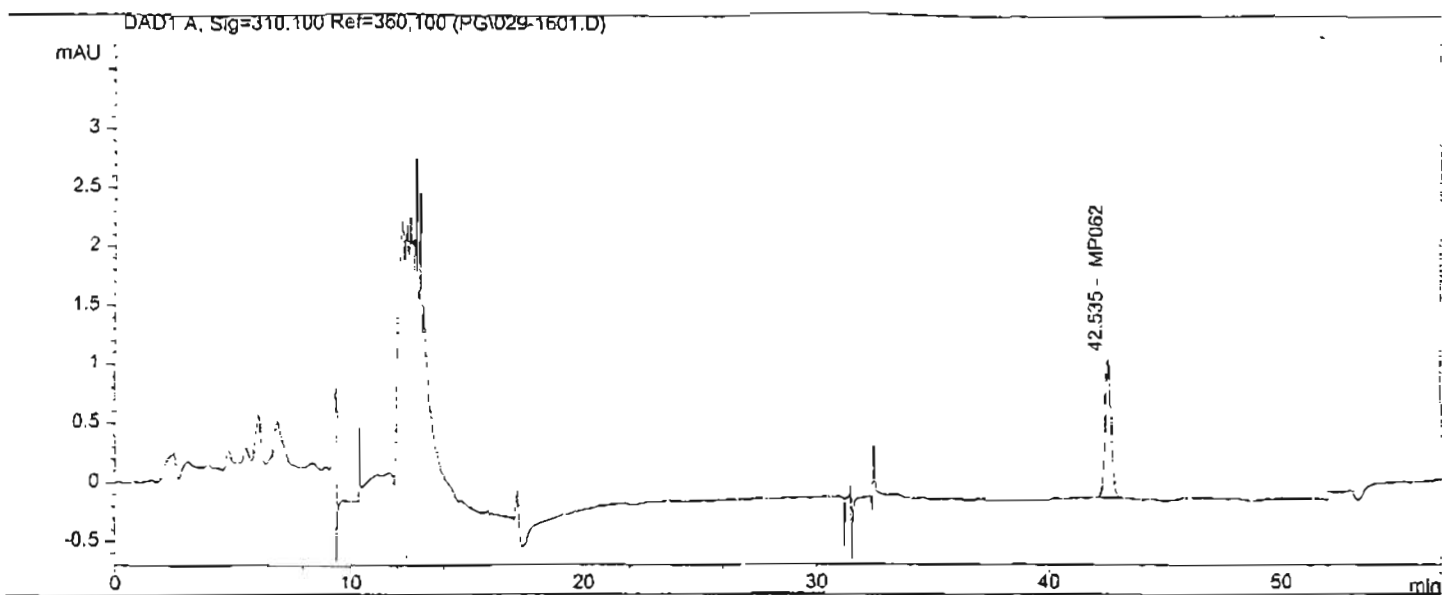
Analytical Enforcement Method (HPLC/Column Switching/UV) for the Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name :MP062 STD(0.525)
 Sample Info. :STD. MP062--0.525 ug/ml

Injection Date :6/25/1998 10:57:29 PM Seq Line : 18
 Acq. Method :DUPONT3.M Vial No. : 31
 Inj. number: 2
 Acq Operator :E. Hayes/P. Golden Inj. Vol : 100 µl
 Analysis Method :C:\HPCHEM\1\METHODS\DUPONT3.M

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	42.498	BB	0.292	1.729	32.536	MP062



=====
 B98-(37-46) DPX-MP062 TMV (Linearity Study)
 =====

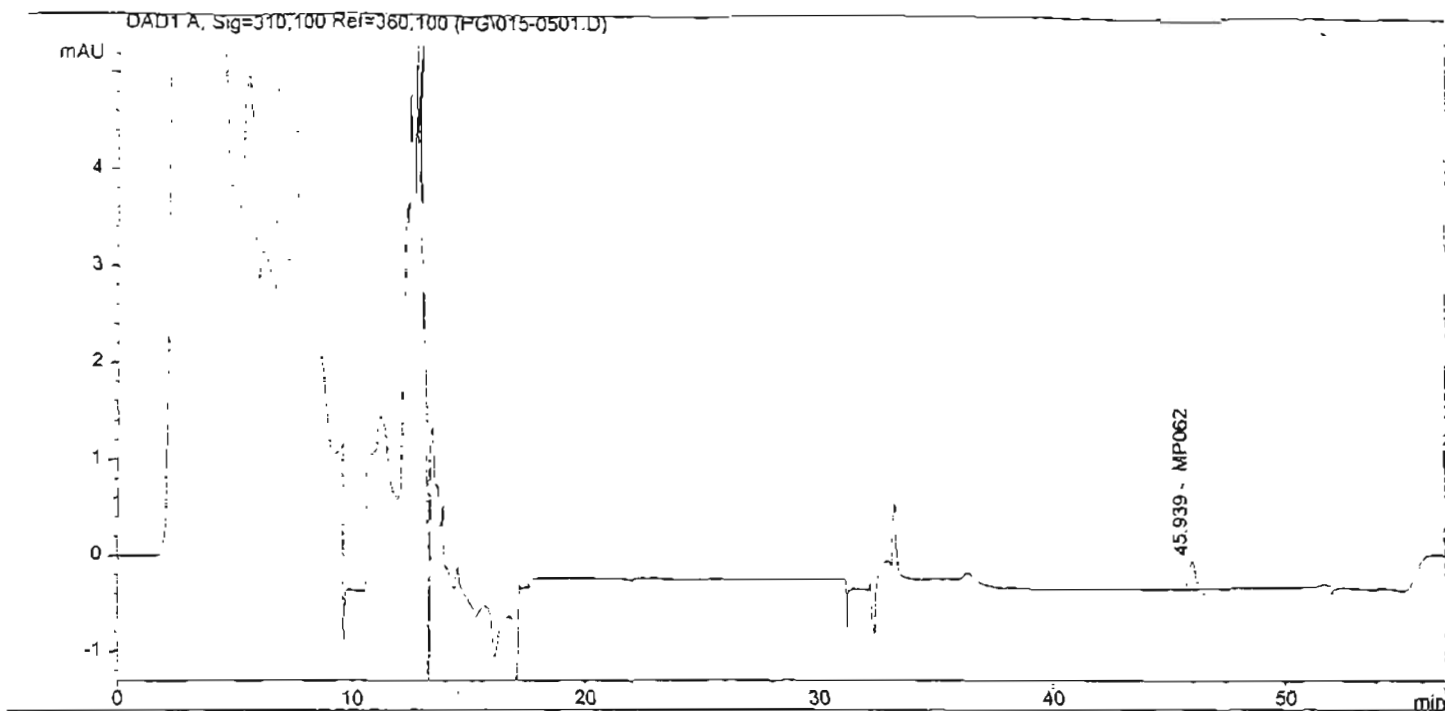
Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name :Cott-S 3.0-1
 Sample Info. :CottonSeed Oil, 3.0-1 ppm, 10g/100 ml final volume

Injection Date	:6/25/1998 06:01:14 PM	Seq Line	: 16
Acq. Method	:DUPONT3.M	Vial No.	: 29
		Inj. number:	1
Acq Operator	:E. Hayes/P. Golden	Inj. Vol	: 100 µl
Analysis Method	:C:\HPCHEM\1\METHODS\DUPONT3.M		

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	42.535	BB	0.287	1.187	21.872	MP062



=====
 B98-(37-46) DPX-MPC62 TMV (Linearity Study)
 =====

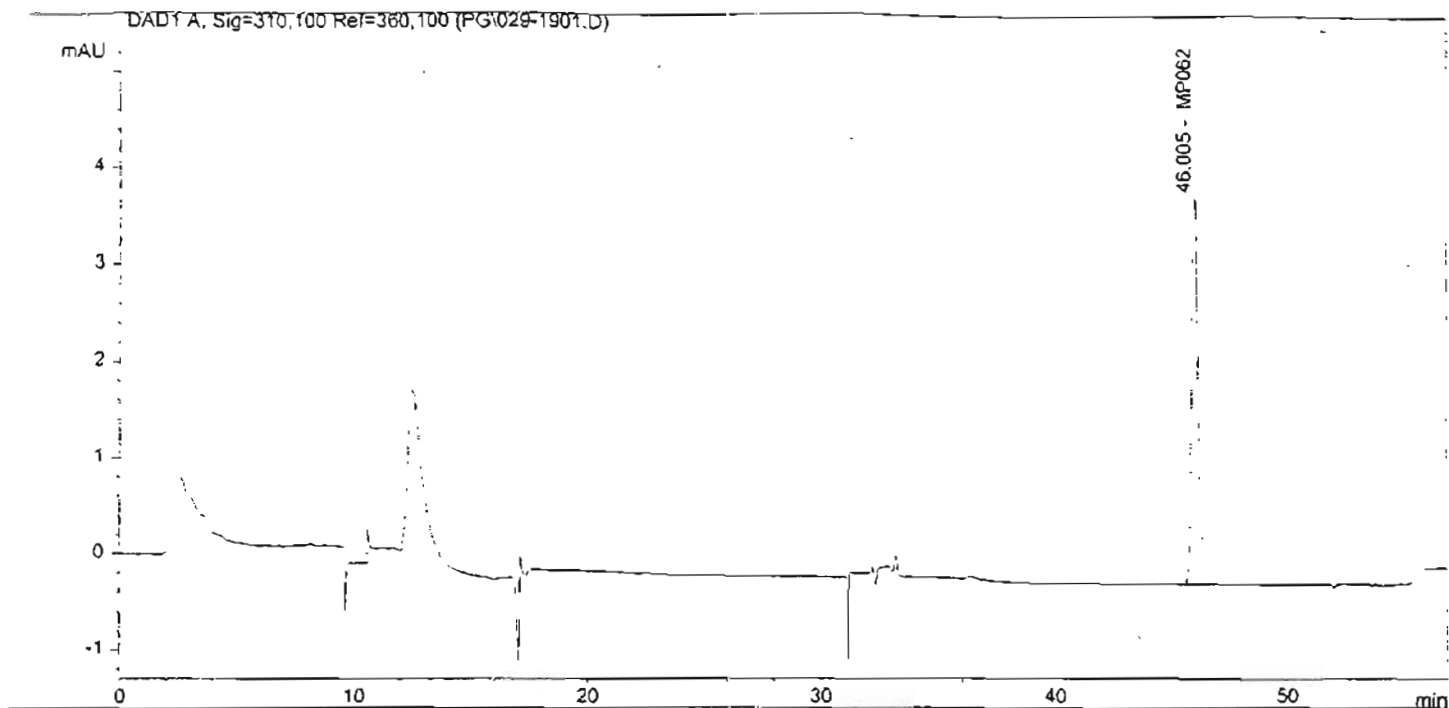
Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name : Tomatoes-0.01-1
 Sample Info. : Tomatoes, 0.01-1 ppm spike, 10g/1.5 ml final volume

Injection Date	: 08/27/1998 20:18:32 PM	Seq Line	: 5
Acq. Method	: DUPONT3.M	Vial No.	: 15
		Inj. number:	: .1
Acq Operator	: Elmer H. Hayes	Inj. Vol	: 100 µl
Analysis Method	: C:\HPCHEM\1\METHODS\DUPONT3.M		

Signal 1 : DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	45.939	PB	0.296	0.296	5.638	MP062



=====
 B98-(37-46) DPX-MP062 TMV (Linearity Study)
 =====

Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

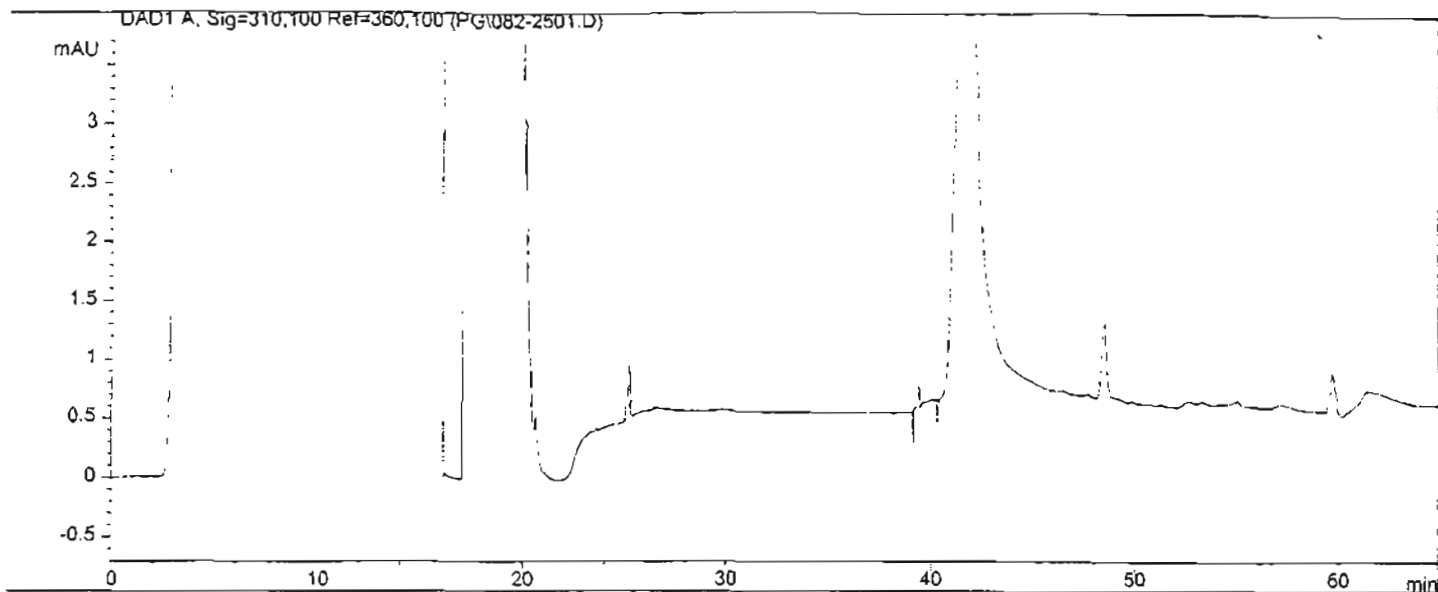
Sample Name :MP062 STD(0.875)
 Sample Info. :STD. MP062--0.875 ug/ml

Injection Date :09/02/1998 16:20:07 PM
 Acq. Method :DUPONT3.M
 Acq Operator :Elmer H. Hayes
 Analysis Method :C:\HPCHEM\1\METHODS\DUPONT3.M

Seq Line : 19
 Vial No. : 29
 Inj. number: 1
 Inj. Vol : 100 µl

Signal 1 :DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	46.005	BB	0.294	4.004	76.731	MP062



B98-(37-46) DPX-MP062 TMV

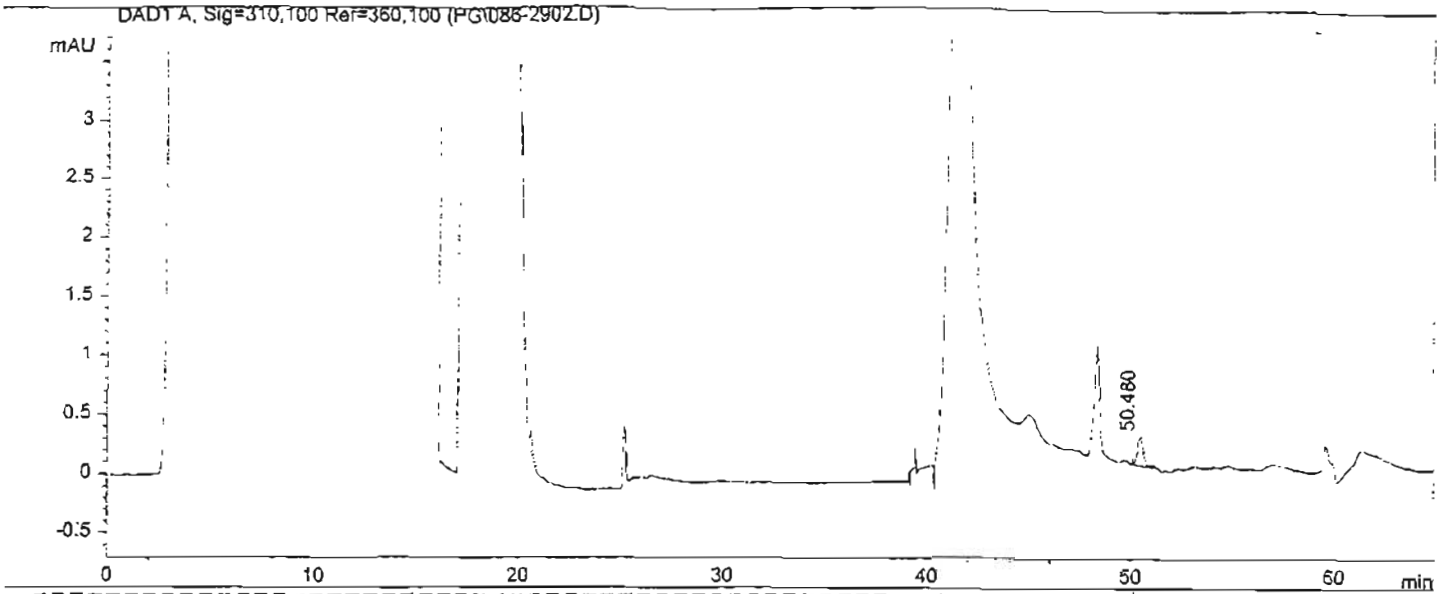
Analytical Enforcement Method (HPLC/Column Switching/UV) for the Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name : Corn Control -1
 Sample Info. : Control Sweet Corn-1
 # 1 sample--10g/1.5 ml final volume

Injection Date : 7/29/1998 17:26:51 PM Seq Line : 25
 Acq. Method : DUPONT3.M Vial No. : 82
 Inj. number: 1
 Acq Operator : E. Hayes/ J.F. Negrón Inj. Vol : 100 µl
 Analysis Method : C:\HPCHEM\1\METHODS\DUPONT3.M

Signal 1 : DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
--------	----------	------	-------------	--------	------	------



B98-(37-46) DPX-MP062 TMV

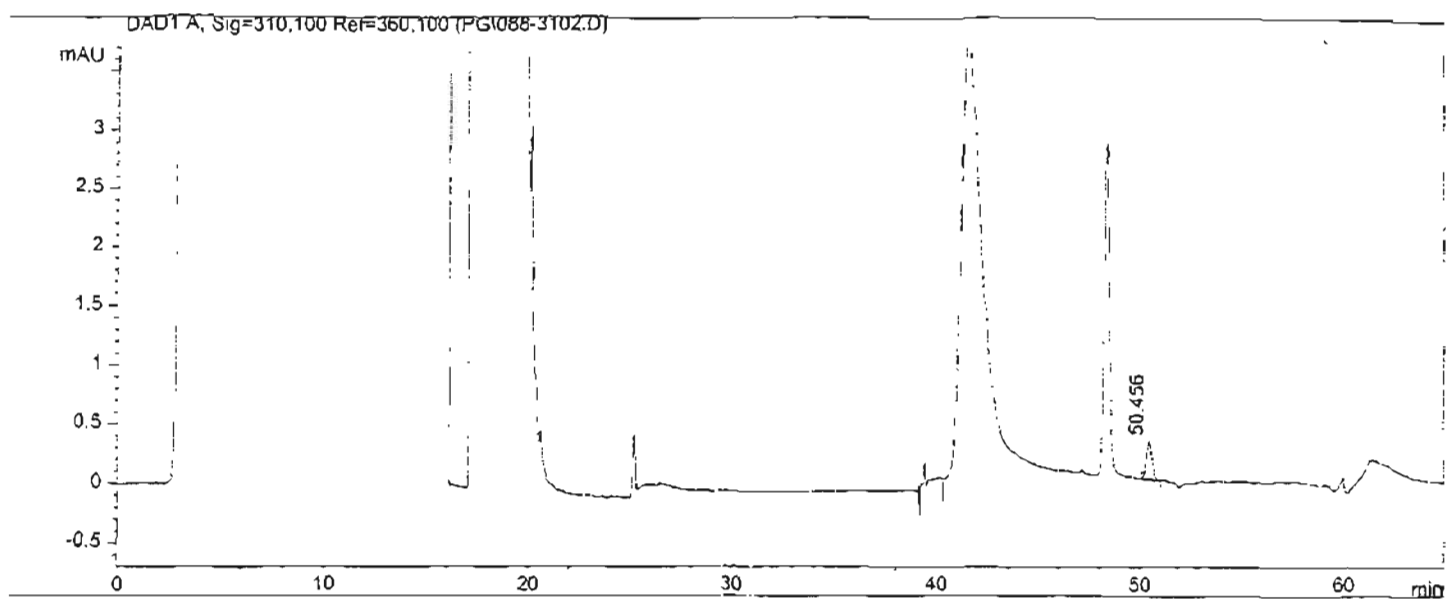
Analytical Enforcement Method (HPLC/Column Switching/UV) for the Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name : Corn 0.01-2
Sample Info. : Sweet Corn, 0.01-2 ppm spike, 10g/1.5ml final volume

Injection Date : 7/30/1998 03:31:37 PM Seq Line : 29
 Acq. Method : DUPONT3.M Vial No. : 86
 Acq Operator : E. Hayes/ J.F. Negron Inj. number: 2
 Analysis Method : C:\HPCHEM\1\METHODS\DUPONT3.M Inj. Vol : 100 µl

Signal 1 : DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	50.480	BB	0.312	0.244	4.934	



B98-(37-46) DPX-MP062 2MV

=====
 Analytical Enforcement Method (HPLC/Column Switching/UV) for the
 Determination of Residues of DPX-KN128 and IN-KN127 in Crops.

Sample Name : Corn 0.02-1
 Sample Info. : Sweet Corn, 0.02-1 ppm spike, 10g/1.5ml final volume

Injection Date : 7/30/1998 08:00:26 PM Seq Line : 31
 Acq. Method : DUPONT3.M Vial No. : 88
 Inj. number: 2
 Acq Operator : E. Hayes/ J.F. Negron Inj. Vol : 100 µl
 Analysis Method : C:\HPCHEM\1\METHODS\DUPONT3.M

Signal 1 : DAD1 A, Sig=310,100 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Height	Area	Name
1	50.456	BB	0.298	0.326	6.310	



13544

002076

Chemical: Invalid PC Code

PC Code: 067710
HED File Code: 11000 Chemistry Reviews
Memo Date: 10/05/99
File ID: DPD257972
Accession Number: 412-01-0056

HED Records Reference Center
12/05/2000