

US EPA ARCHIVE DOCUMENT

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- 1.2.4 The methanol is evaporated from this aliquot using a 90°C water bath and the aqueous portion remaining is cleaned up using a solid phase extraction column (SPE).
- 1.2.5 Norflurazon and desmethyl norflurazon are eluted from the column with dichloromethane, solvent exchanged into toluene and quantitated by gas chromatography using ⁶³Ni electron capture detector (EC) and a megabore methyl silicone capillary column (30 m x 0.53 mm (i.d.), 0.88 µm film thickness).

2. SAFETY

- 2.1 The oral LD₅₀ of technical norflurazon in rats is greater than 9000 mg/kg body weight. The dermal toxicity (LD₅₀ in rabbits) is greater than 20000 mg/kg. Norflurazon is not a skin irritant, skin sensitizer or an eye irritant.
- 2.2 Normal laboratory precautions are adequate for safe handling of norflurazon. Normal first aid procedures are appropriate for exposure to norflurazon, (wash with soap and water for skin contact, flush eyes thoroughly with water for eye contact and induce vomiting for accidental ingestion).
- 2.3 Dichloromethane, pentane, methanol and toluene are flammable and should not be used near heat, sparks or open flames.
- 2.4 All solvents should be used in well ventilated laboratories.
- 2.5 Protective gloves should be worn during extraction and clean-up procedures. Safety glasses should be worn at all times.
- 2.6 Potassium hydroxide is a strong caustic agent and should be immediately washed off with large volumes of water after any accidental contact.
- 2.7 Disposal of sample extracts and standard solutions must be done in compliance with on-site policy and procedures.

3. MATERIALS AND METHODS

3.1 Apparatus

- 3.1.1 Analytical balance.
- 3.1.2 Pipets, graduated, 0.2, 0.5 and 1-mL, Pyrex.
- 3.1.3 Volumetric Flasks, 100-mL, 5-mL.
- 3.1.4 Pipets, pasteur, 9 inch disposable.
- 3.1.5 Top loading balance.

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- 3.1.6 Dilution bottles, glass 150-mL.
- 3.1.7 Homogenizer, Brinkman, Polytron®.
- 3.1.8 Water bath, 90 ± 2°C.
- 3.1.9 Bottles, glass, 8-oz with Poly-Seal screw-caps.
- 3.1.10 Shaker, platform, Eberbach Company, Ann Arbor, Michigan.
- 3.1.11 Funnels, Buchner, 7.0 cm.
- 3.1.12 Filter paper, 7.0 cm Whatman A.
- 3.1.13 Flasks, suction filter 250 and 500-mL with side arm.
- 3.1.14 Cylinders graduated, glass, 100, 200, and 250-mL
- 3.1.15 Concentrator, Kuderna Danish (KD), 125-mL.
- 3.1.16 Distillation receiver tubes (KD tubes), 12-mL.
- 3.1.17 Reservoir tube, 15-mL, Analytichem International, Harbor City, CA; Catalog No. 601500.
- 3.1.18 Solid Phase Extraction Columns, Bond Elute® C-18, (3-mL, 500 mg). Analytichem International Harbor City, CA (Catalog #607303).
- 3.1.19 Vac Elut Processing station, Analytichem International Harbor City, CA; Catalog No. AI6000.
- 3.1.20 Evaporator, N-Evap with 40°C water bath - (Organomation Assoc., South Berlin MA. 01549-0159)
- 3.1.21 Gas chromatograph, HP-5880 A equipped with ⁶³Ni electron capture detector, HP 7673A autosampler and HP 5880A series level four GC terminal.
- 3.1.22 Column, capillary, HP megabore methyl silicone gum (SE-30), 30m x 0.53 mm (i.d.) x 0.88 µm film thickness.
- 3.1.23 Data system, Varian CDS 401.
- 3.1.24 RS1 software, BBN Software Products Corp., 10 Fawcett Street, Cambridge, MA. 02238
- 3.1.25 VAX Model 750 Computer, or equivalent.

3.2 Reagents

- 3.2.1 Methanol, high purity-solvent, B & J Muskegon, MI.

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- 3.2.2 Toluene, Baker Resi-Analyzed, J.T. Baker Company, Phillipsbury, N.J.
- 3.2.3 Potassium hydroxide, reagent grade.
- 3.2.4 Celite powder, J. T. Baker Chemical Co., Phillipsburg, N.J. 08865.
- 3.2.5 Boiling chips, 10 mesh, Hengar Company, Philadelphia, PA.
- 3.2.6 Deionized Water, Milli-Q water Purification System, Millipore Corporation, Bedford, Mass.
- 3.2.7 Pentane, Baker Resi-Analyzed, J.T. Baker Company, Phillipsbury, N.J.
- 3.2.8 Dichloromethane, Baker Resi-Analyzed, J.T. Baker Company, Phillipsbury, N.J.

3.3 Preparation of Standard Solutions

- 3.3.1 Norflurazon: 4-chloro-5-(methylamino)-2-(α,α,α -trifluoro-m-tolyl)-3(2H)-pyridazinone and desmethyl norflurazon: 4-chloro-5-amino-2-(α,α,α -trifluoro-m-tolyl)-3(2H)-pyridazinone; Sandoz Crop Protection Analytical Reference Standards. See Figure 1 for molecular structures.
- 3.3.2 Weigh 100.0 mg each of norflurazon and desmethyl norflurazon analytical reference standard in a 100-mL volumetric flask. Dissolve the standards and bring to the mark with methanol. The concentration of this stock solution is 1×10^{-6} g/ μ L for each compound.
- 3.3.3 Transfer 1.0 mL of the 1×10^{-6} g/ μ L stock solution from 3.3.2 to a 100-mL volumetric flask and bring to the mark with methanol. This solution (1×10^{-8} g/ μ L) is used for fortifying check samples for recovery determination and for the preparation of GC standard solutions.
- 3.3.4 Prepare a range of GC standards by diluting the 1×10^{-8} g/ μ L fortification standard from 3.3.3 to 100 mL (in a 100-mL volumetric flask) with toluene as follows:

<u>Volume of 1×10^{-8} g/μL standard solution</u>	<u>Concentration of final solution</u>
1.0 mL	1.0×10^{-10} g/ μ L
0.5 mL	5.0×10^{-11} g/ μ L
0.20 mL	2.0×10^{-11} g/ μ L
0.05 mL	5.0×10^{-12} g/ μ L
0.025 mL	2.5×10^{-12} g/ μ L

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3.4 Extraction Procedure

- 3.4.1. Weigh 10g of chopped/pulverized sample into a 150-mL dilution bottle.
- 3.4.2 For recovery determinations add 0.05 mL or 0.10 mL of the 1×10^{-8} g/ μ L fortification standard solution to the 10 g check sample giving a 0.05 ppm or 0.10 ppm fortification, respectively.
- 3.4.3 Add 70 mL of aqueous 0.5 N KOH (100 mL for bulky samples like hay, cottonseed and peanut hulls) to the dilution bottle and homogenize with a Polytron® for 0.5 to 1.0 minute at medium speed.
- 3.4.4 Rinse the Polytron blade after each use with 10 mL of aqueous 0.5 N KOH and collect the rinse in the dilution bottle.
- 3.4.5 Loosely cover the dilution bottle with aluminum foil and set the dilution bottle into a 90°C water bath for 1 hour. The level of water in the bath should cover the level of liquid in the dilution bottle.
- 3.4.6 Cool the bottle to room temperature, mix the contents by swirling and transfer the sample to an 8-oz glass bottle. Before transferring a bulky sample (hay or peanut hulls), add 25 mL of methanol to the dilution bottle and mix with a spatula.
- 3.4.7 Rinse the dilution bottle with 4 x 25 mL methanol (3 x 25 mL of methanol for bulky samples to which 25 mL of methanol was already added in Section 3.4.6) and add these rinses to the 8-oz glass bottle.
- 3.4.8 Tightly cap the 8-oz glass bottle and shake for 15 minutes on a platform shaker.
- 3.4.9 Suction filter the extract through a Buchner funnel fitted with a filter paper covered by a thin layer (about 0.25 cm) of celite into a 250-mL suction filter flask. Use a 500-mL suction filter flask for oil nut samples.
Caution! Instantaneous foaming will occur in the suction filter flask during filtration. Control the vacuum to avoid loss of filtrate due to overflow through the side arm of the suction filter flasks.
- 3.4.10 Rinse the 8-oz glass bottle with 2 x 10 mL methanol and pass these rinses through the Buchner funnel into the suction filter flask.

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- 3.4.11 Transfer the filtrate to a 200-mL or 250-mL graduated cylinder and adjust the volume of the filtrate to 200 mL with methanol rinses of the suction filter flask.
- 3.4.12 Transfer 20 mL (1 gram equivalent) of the extract (filtrate) to a 125-mL KD apparatus (see Sections 3.1.15 and 3.1.16) and add 2-3 boiling chips to the KD tube. Do not attach any condenser to the KD flask.
- 3.4.13 Evaporate the methanol using a 90°C water bath until no further condensate is observed in the neck of the KD flask. The final volume will be about 7-10 mL depending on sample type (Section 3.4.3).

3.5 Cleanup by Solid Phase Extraction (SPE)

- 3.5.1 Attach a 15-mL reservoir tube (Section 3.1.17) to a 3-mL, (500 mg) C-18 Bond Elut SPE column (Section 3.1.18) and attach the setup to a VAC Elut Processing station (Section 3.1.19).
- 3.5.2 Apply a low vacuum of about 5 PSI to the processing station.
- 3.5.3 Pass sequentially 5 mL of methanol and 5 mL of deionized water through the column to wash and condition it.
- 3.5.4 Add the aqueous sample extract (filtrate) from section 3.4.13 to the C-18 column setup. Adjust the vacuum to get a flow rate of about 2 drops per second. Do not let the column go dry until Section 3.5.6.
- 3.5.5 Wash the column sequentially with 10 mL of 25% methanol/H₂O and 10 mL of pentane.
- 3.5.6 Release the vacuum and detach the reservoir from the column.
- 3.5.7 Remove water drops if any from the top of the column bedding by gently inserting a piece of tissue paper.
- 3.5.8 Reapply a vacuum of about 10 PSI to the processing station and let the column dry for 5 minutes. Release the vacuum and place a rack holding a 5-mL volumetric flask inside the Vac-Elut.
- 3.5.9 Align the 5-mL volumetric flask under the column as you reattach the cover, making sure the tube on the bottom of the processing station cover underneath the column is in the volumetric flask.
- 3.5.10 Apply a vacuum of about 10 PSI to the processing station and elute norflurazon and desmethyl

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norflurazon with 2 x 1-mL aliquots of dichloromethane, collecting both eluates in the 5-mL volumetric flask.

- 3.5.11 Transfer the dichloromethane to a K-D tube. Rinse the volumetric flask with 2 x 1-mL aliquots of dichloromethane and add these rinses to the KD tube.
- 3.5.12 Place the KD tube in a 40°C water bath and evaporate the dichloromethane to dryness under a gentle stream of nitrogen.
- 3.5.13 Redissolve the residue in 2 mL of toluene. The sample extract is now ready for gas chromatographic analysis.

3.6 Analysis

3.6.1 Gas Chromatographic Conditions

The following instrument and conditions are suitable for the analysis of norflurazon and desmethyl norflurazon in raw agricultural commodities. Other conditions may be used provided that the subject compounds are separated from sample interferences and the detector response is linear over the range of interest. Verify that the detector response is linear over the desired range and the retention time is stable, on a daily basis. This is preferably done by injecting standard solutions prior to the analysis of samples and after each 2-4 samples during analysis.

3.6.1.1 Gas Chromatograph: Hewlett-Packard model 5880A equipped with a HP 7673A autosampler and HP level 4 integrator. The GC was interfaced with a Varian CDS 401 chromatography data system.

3.6.1.2 Column: Megabore, capillary, HP-1 crosslinked methyl silicone gum (SE-30) FSOT, 30 m x 0.53 mm (id), 0.88 µm film thickness.

3.6.1.3 Conditions:

Oven Temperature: 220 °C, for 6.5 min.
Post Temperature: 250 °C for 5 min.
Equilibration Time: 1 minute
Injector Temperature: 220 °C
Detector Temperature: 350 °C
Carrier gas: helium at 11 mL/min.
Makeup gas: 5% argon/methane at 30 mL/min.
Chart speed: 1 cm/min.
Retention times:
Norflurazon = 4.20 min.

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Desmethyl Norflurazon = 4.82 min.

3.6.2 Quantitation

- 3.6.2.1 Prepare a standard curve for each compound by injecting GC standards (Section 3.3.4) of known concentrations and plotting peak heights versus concentration of injected standards, on log-log paper, (Inject 2 μL aliquots of each of the standards from section 3.3.4).
- 3.6.2.2 Determine the concentrations of norflurazon and desmethyl norflurazon in a 2 μL injected aliquot of sample from their peak heights and the standard curves generated in section 3.6.2.1.
- 3.6.2.3 Calculate the concentration of the residue in the sample using the following expression:

$$\text{PPM (ng/mg)} = \frac{C(\text{ng}/\mu\text{L}) \times V(\mu\text{L})}{W(\text{mg})}$$

Where;

ppm = concentration of compound in the sample in parts per million (ng/mg).

C = Concentration of compound in the injected aliquot, determined from the standard curve, (in nanograms per microliter).

V = Final volume of the sample extract (in microliters) taking into consideration all dilutions.

W = Weight of sample taken for analysis, in milligrams.

3.6.3 Quantitation Using RS1 Computer Software and VAX Model 750 Computer

- 3.6.3.1 Inject 2 μL aliquots each of the standards from Section 3.3.4 and of each sample extract from Section 3.5.14.
- 3.6.3.2 Prepare separate tables of standards and samples according to the formats of Tables 1 and 2 of the Appendix.
- 3.6.3.3 Use #ppm, an RS-1 procedure, for calculating

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the levels of analytes in the sample extracts. The variables of the procedures are given in the Appendix.

- 3.6.3.4 Supply the names for the graph (Standard Curve) and the results table.
- 3.6.3.5 Set globals as described in Table 3 of the Appendix, for printing graph and results table.
- 3.6.3.6 The results table gives the concentration (ppm) of analytes in the sample.
- 3.6.3.7 Printout the tables and graph on a printer by using "print" command followed by the name of the table or graph given in Section 3.6.3.4.

3.7 Interferences

3.7.1 Sample Matrices

Several crops of considerably different makeup have been analyzed using this procedure. New matrices not included in this report will have to be validated before this method is used to quantitate residues.

3.7.2 Solvents and Labware

Interferences have not been a problem when using the high quality solvents listed under Section 3.2 and carefully cleaned labware.

3.8 Time Required for Analysis

A single sample can be extracted and cleaned up in four hours for gas chromatographic analysis. Ten orange samples were extracted, cleaned up and analyzed by gas chromatography in two eight hour days.

4. RESULTS AND DISCUSSION

4.1 Accuracy

Recoveries of subject compounds from fortified raw agricultural commodity check samples are listed in Table 1. Average recoveries ($\bar{x} \pm sd$) for norflurazon ranged from 92.3 % \pm 9.9 % (peanut green hay) to 102 % \pm 4.0 % (grape fruit). Average recoveries for desmethyl norflurazon ranged from 87.0 % \pm 9.6 % (peanut green hay) to 104.0 % \pm 4.0 % (peanut hulls).

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4.2 Precision

The coefficient of variation for the recoveries in Table 1 are from 1.5 % to 11.8 % for norflurazon and from 2.9 % to 12.6 % for its desmethyl metabolite.

4.3 Limit of Detection

The limit of detection was 0.01 ppm for desmethyl norflurazon in all matrices tested. For norflurazon the limit of detection was 0.025 for peanut hay (dry and green) and 0.01 ppm for all other matrices tested. Recoveries from fortifications as low as 0.05 ppm were acceptable. No significant difference in recovery was observed at different fortification levels.


5. CONCLUSIONS

This method was developed by Sandoz Crop Protection for the determination of norflurazon herbicide and its desmethyl metabolite in raw agricultural commodities. The method is useful for detecting and quantitating residues of these compounds in raw agricultural commodities at or above the limit of detection of 0.025 or 0.01 ppm as described in Section 4.3. The recoveries of subject compounds from fortified raw agricultural commodities are generally higher than 90% for both compounds.

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6. CERTIFICATION

I hereby state as principal author and chemist of record that the method described herein was conducted within the framework of the GLP program at Sandoz Crop Protection. The description of this method and the supporting data (recoveries) are accurate and correct to the best of my knowledge.

<u>SYED ALI</u>	<u></u>
name	signature
<u>SENIOR SCIENTIST</u>	<u>6/10/88</u>
title	date

I hereby state as the study Director for the analysis and reporting portion of the above method that the contents herein are accurate and correct to the best of my knowledge.

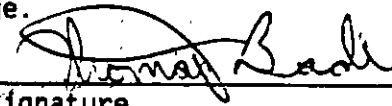
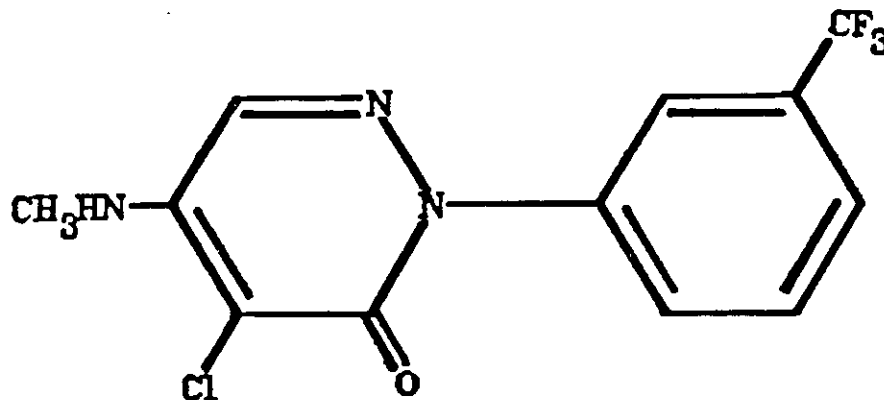
<u>Thomas Bado</u>	<u></u>
name	signature
<u>Section header</u>	<u>6/10/88</u>
title	date

Table 1. Recoveries Of Norflurazon And Desmethyl Norflurazon From Fortified Check Samples Of Various Raw Agricultural Commodities (RAC).

RAC	Level of Fortification	Replication	Percent Recoveries	
			Desmethyl Norflurazon	Norflurazon
1. Asparagus	0.1	1	86	99
	0.1	2	98	109
	0.1	3	94	104
	0.1	4	80	89
	0.1	5	87	88
	0.1	6	84	91
			$\bar{x} \pm s.d.$	88.2 ± 6.6
2. Grapefruit	0.05	1	102	102
	0.05	2	110	106
	0.05	3	86	98
		$\bar{x} \pm s.d.$	99.3 ± 12.2	102.0 ± 4.0
3. Lemon	0.05	1	78	92
	0.05	2	100	102
	0.05	3	94	100
		$\bar{x} \pm s.d.$	90.1 ± 11.4	98.0 ± 5.3
4. Orange	0.05	1	96	96
	0.05	2	92	94
	0.05	3	96	96
	0.05	4	94	96
	0.05	5	100	98
		$\bar{x} \pm s.d.$	95.6 ± 3.0	96.0 ± 1.4
5. Peanut green hay	0.1	1	80	92
	0.1	2	98	114
	0.1	3	83	95
		$\bar{x} \pm s.d.$	87.0 ± 9.6	100.3 ± 11.9
6. Peanut dry hay	0.1	1	90	99
	0.1	2	85	97
	0.1	3	87	81
		$\bar{x} \pm s.d.$	87.3 ± 2.5	92.3 ± 9.9
7. Peanut hull	0.1	1	100	98
	0.1	2	104	101
	0.1	3	108	104
		$\bar{x} \pm s.d.$	104.0 ± 4.0	101.0 ± 3.0
8. Peanut meat	0.1	1	93	106
	0.1	2	84	95
	0.1	3	102	97
		$\bar{x} \pm s.d.$	93.0 ± 9.0	99.3 ± 5.9

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Norflurazon



desmethyl Norflurazon

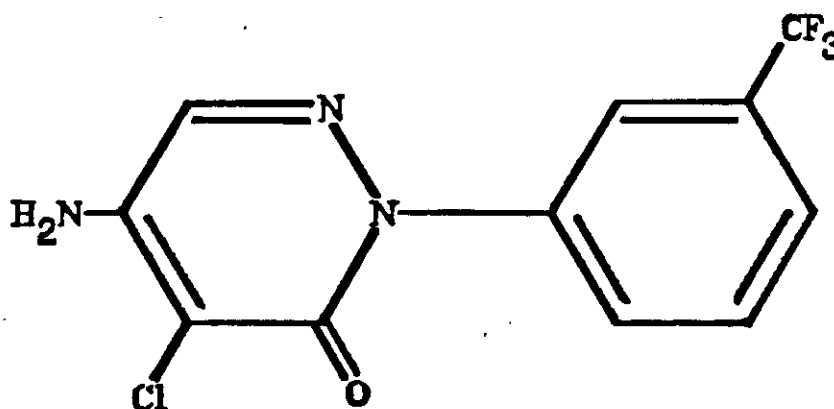


Figure 1. Chemical Structures of Norflurazon: 4-chloro-5-(methylamino)-2-(α,α,α -trifluoro-m-tolyl)-3(2H)-pyridazinone and Desmethyl Norflurazon: 4-chloro-5-amino-2-(α,α,α -trifluoro-m-tolyl)-3(2H)-pyridazinone;

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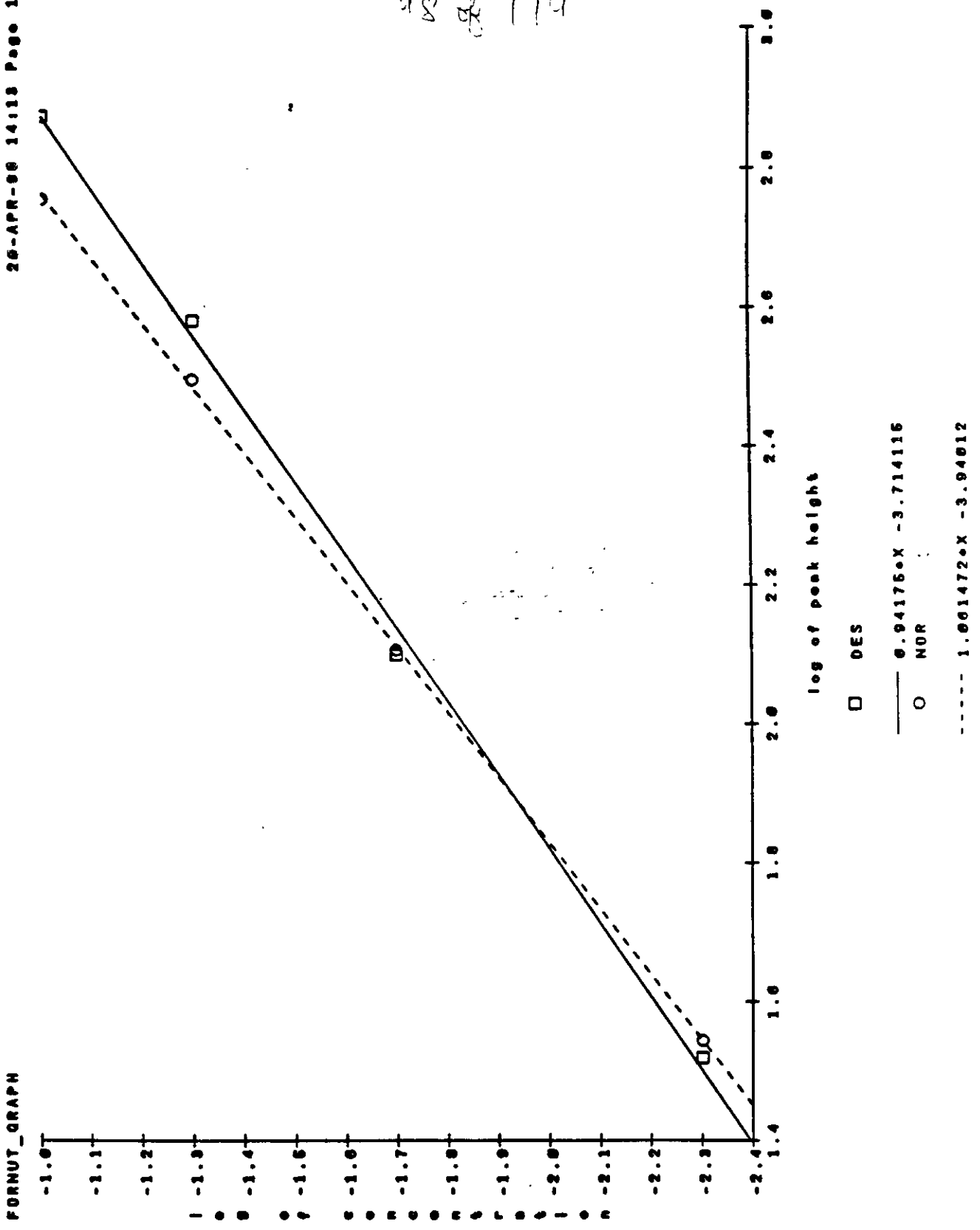
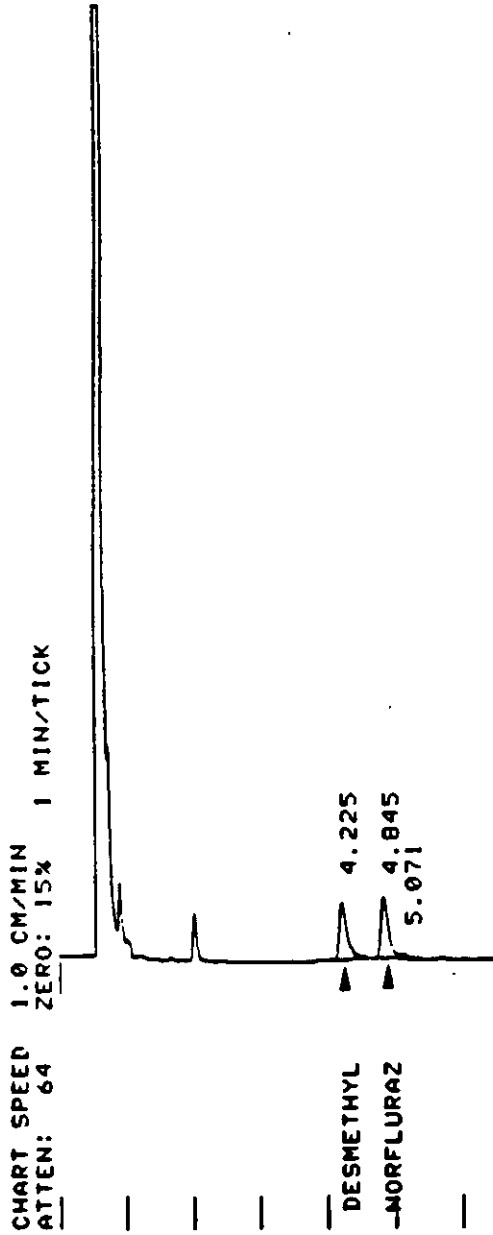


Figure 2. Computer Generated Calibration Curve for the determination of Norflurazon (solid line) and Desmethyl norflurazon (dashed line). The standards were interspersed with samples during analysis.

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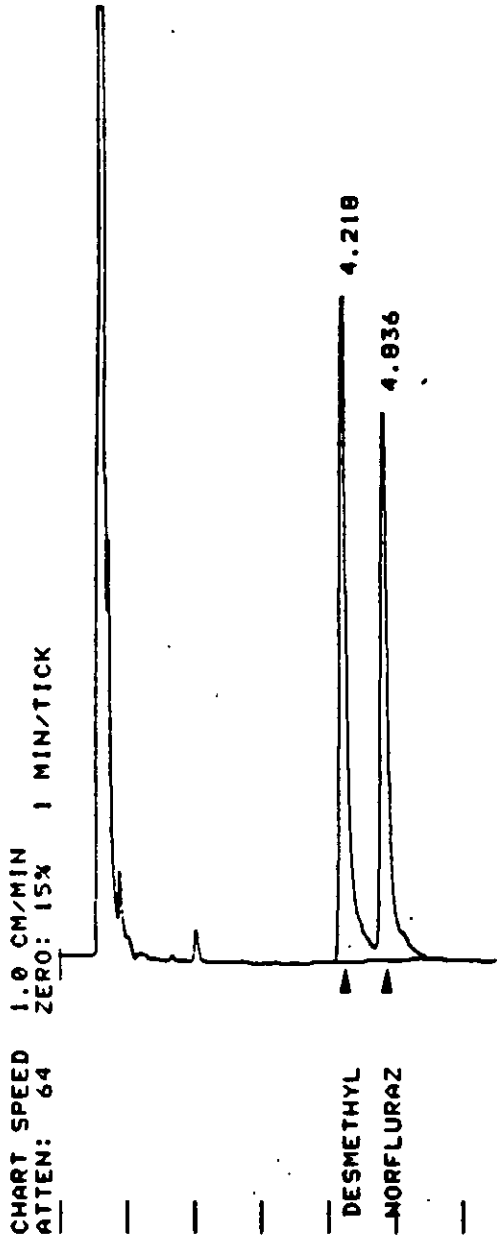
TITLE: NORF+DES IN PEANUTS 11:08 19 APR 88
CHANNEL NO: 1 SAMPLE: 5X10-12 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT FACTOR	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.607533	4.225	0.005	3292	BB	6.70
2	NORFLURAZ	0.564972	4.845	0.005	3540	BV	6.55
TOTALS:					6832		

UNIDENT AREA: 231
DETECTED PKS: 3 REJECTED PKS: 0
AMT STD: 0.20000
NOISE: 85.0 OFFSET: -54
ERRORS:
FACTOR NOT UPDATED

Figure 3. Representative EC-GC chromatogram of 0.01 ng each of norflurazon and desmethyl norflurazon. A 2 µL injection of a 0.005 ng/µL standard solution.

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TITLE: NORF+DES IN PEANUTS 13:11 19 APR 88

CHANNEL NO: 1 SAMPLE: 5Y10-11 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT FACTOR	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	HI/2 (SEC)
1	DESMETHYL	0.052217	4.218	-0.002	38302	BV	5.90
2	NORFLURAZ	0.063599	4.836	-0.004	31447	VB	6.50

TOTALS: -0.006 69749

UNIDENT AREA: 0

DETECTED PKS: 2 REJECTED PKS: 0

AMT STD: 0.20000

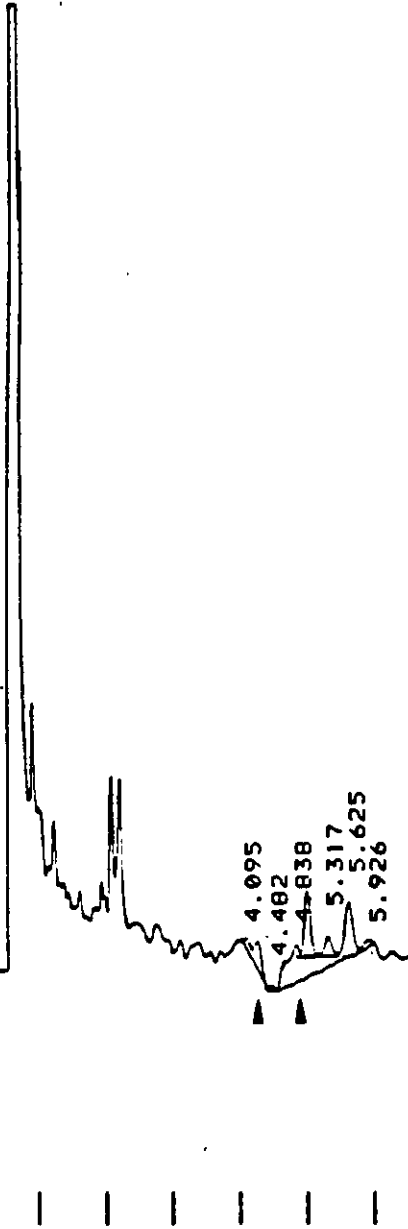
NOISE: 85.0 OFFSET: 25

ERRORS: FACTOR NOT UPDATED

Figure 4. Representative EC-GC chromatogram of 0.1 ng each of norflurazon and desmethyl norflurazon. A 2 µL injection of a 0.05 ng/µL standard solution.

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CHART SPEED 1.0 CM/MIN
ATTEN: 64 ZERO: 15% 1 MIN/TICK



TITLE: NORFLURAZES IN ASPARAGUS 20:05 25 MAR 88

CHANNEL NO: 1 SAMPLE: CK 01427 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	HI/2 (SEC)
2	DESMETHYL	0.0130	4.259	-0.111	1623	VB	3.60
5	NORFLURAZ	0.0533	5.005	-0.015	4897 3774	VV	8.75
TOTALS:					0.0663	-0.126	6520

UNIDENT AREA: 8283

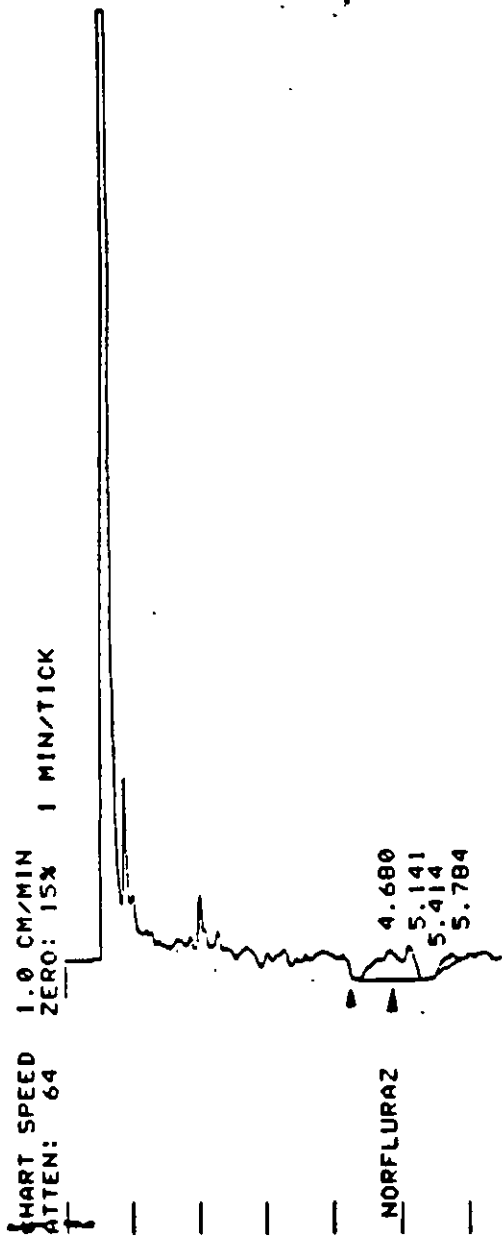
DETECTED PKS: 8 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 35.2 OFFSET: -413

Figure 5. Representative EC-GC chromatogram of an asparagus check sample (86-01427); 1.0 mg equivalents injected; <0.01 ppm each of norflurazon and desmethyl norflurazon detected.

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TITLE: NOPF+DES IN PEANUTS 9:04 19 APR 88

CHANNEL NO: 1 SAMPLE: CK MEAT #93 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	NORFLURAZ	0.0175	4.848	0.008	1686	VV	8.20
TOTALS:		0.0175		0.008	1686		

UNIDENT AREA: 3660

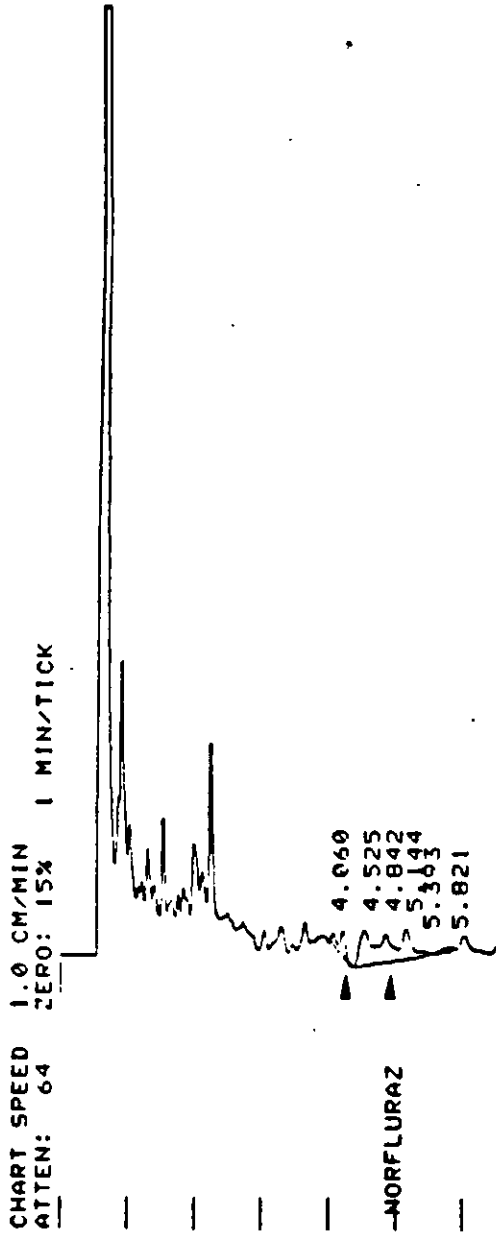
DETECTED PKGS: 5 REJECTED PKGS: 1

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 85.0 OFFSET: -44

Figure 6. Representative EC-GC chromatogram of a peanut meat check sample (86-06793); 1.0 mg equivalents injected; <0.01 ppm each of norflurazon and desmethyl norflurazon detected.

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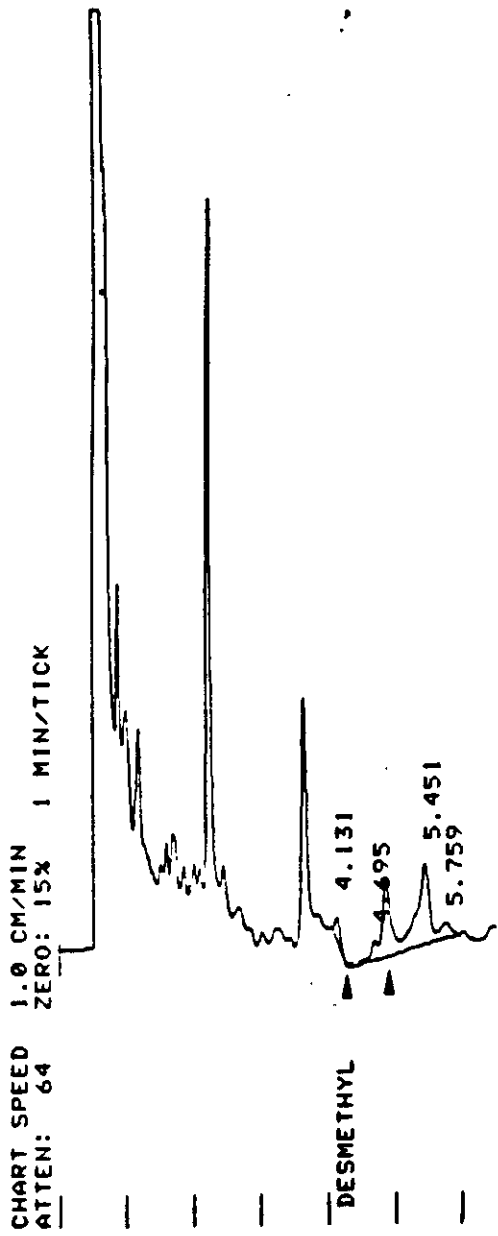


TITLE: NORF+DES IN PEANUTS 9:20 19 APR 88
CHANNEL NO: 1 SAMPLE: CK HULLS 893 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	HI/2 (SEC)
2	DESMETHYL	0.0118	4.206	-0.014	1473	VB	3.45
4	NORFLURAZ	0.0162	4.842	0.002	1560	VV	? 7.90
TOTALS:		0.0280		-0.012	3033		

UNIDENT AREA: 4879
DETECTED PKS: 7 REJECTED PKS: 0
DIVISOR: 1.00000 MULTIPLIER: 1.00000
NOISE: 85.0 OFFSET: -21

Figure 7. Representative EC-GC chromatogram of a peanut hull check sample (86-06793); 1.0 mg equivalents injected; <0.01 ppm each of norflurazon and desmethyl norflurazon detected.



TITLE: NORF+DES IN PEANUTS . 13:04 21 APR 88

CHANNEL NO: 1 SAMPLE: CK GRHAY 84 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.0111	4.131	-0.099	1425	BB	3.70
4	NORFLURAZ	0.0468	4.859	-0.001	4512	VV	8.15
TOTALS:		0.0579		-0.100	5937		

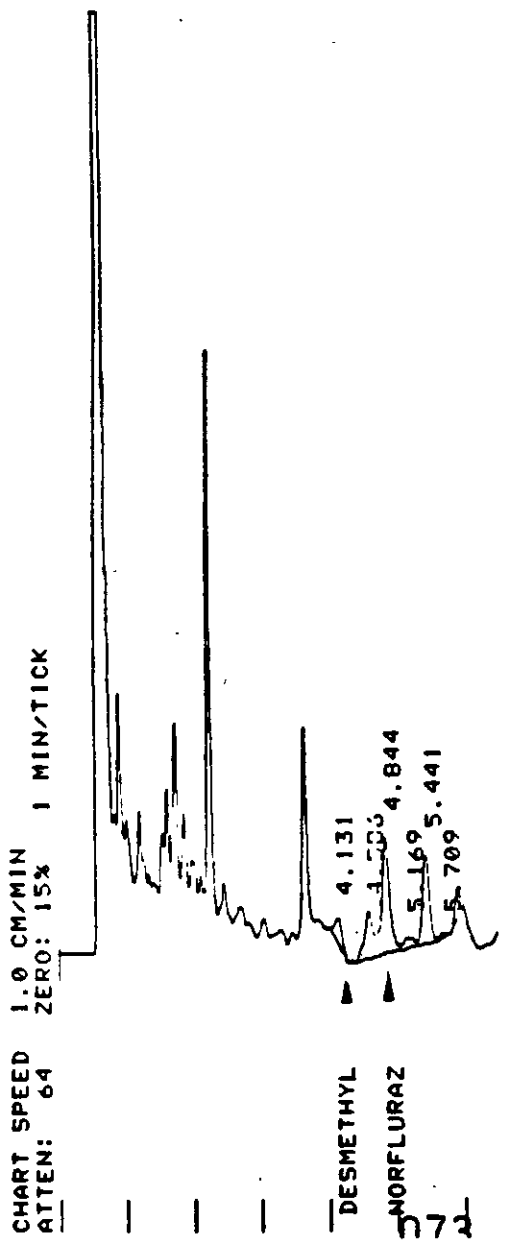
UNIDENT AREA: 6885

DETECTED PKS: 6 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 27.9 OFFSET: 19

Figure 8. Representative EC-GC chromatogram of a peanut green hay check sample (86-06784); 1.0 mg equivalents injected; <0.025 ppm norflurazon and < 0.01 ppm desmethyl norflurazon detected.

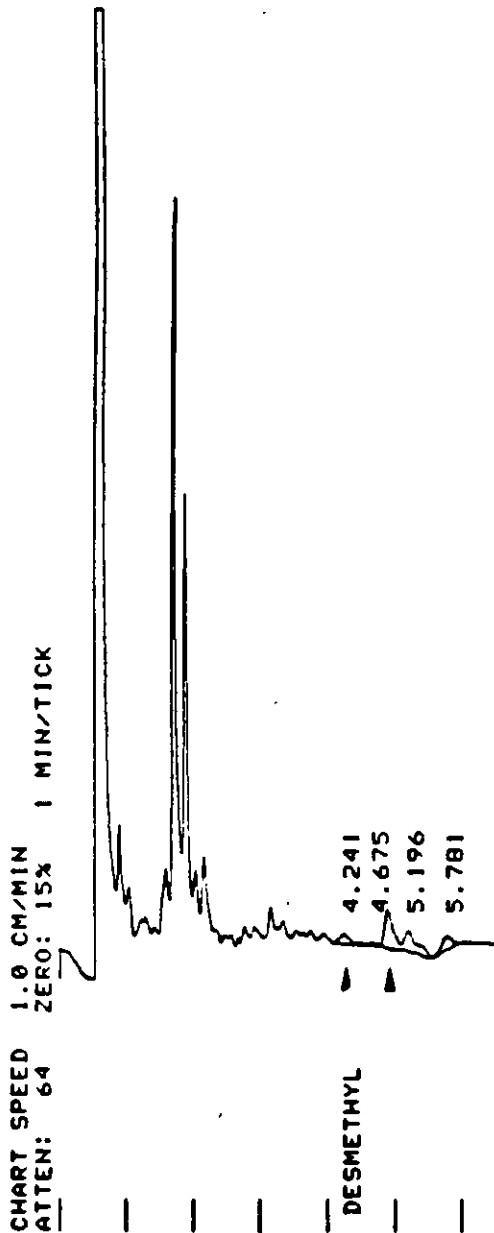


TITLE: NORF+DES IN PEANUTS 9:50 19 APR 88
CHANNEL NO: 1 SAMPLE: CK DR HAY#01 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)	
1	DESMETHYL	0.0116	4.131	-0.089	1446	BB	3.90	
3	NORFLURAZ	0.0692	4.844	0.004	6674	VV	7.15	
TOTALS:					0.0808	-0.085	8120	

UNIDENT AREA: 10143
DETECTED PKS: 8 REJECTED PKS: 1
DIVISOR: 1.00000 MULTIPLIER: 1.00000
NOISE: 85.0 OFFSET: 65

Figure 9. Representative EC-GC chromatogram of a peanut dry hay check sample (86-06801); 1.0 mg equivalents injected; <0.025 ppm norflurazon and <0.01 ppm desmethyl norflurazon detected.



TITLE: NORF+DES IN CITRUS FRUITS 0:19 9 MAY 88

CHANNEL NO: 1 SAMPLE: CK OR 001 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	MI/2 (SEC)
1	DESMETHYL	0.0038	4.241	-0.009	479	BV	7.15
3	NORF	0.0000	4.892	0.012	2139	VV	7.10
TOTALS:					0.0038	2618	

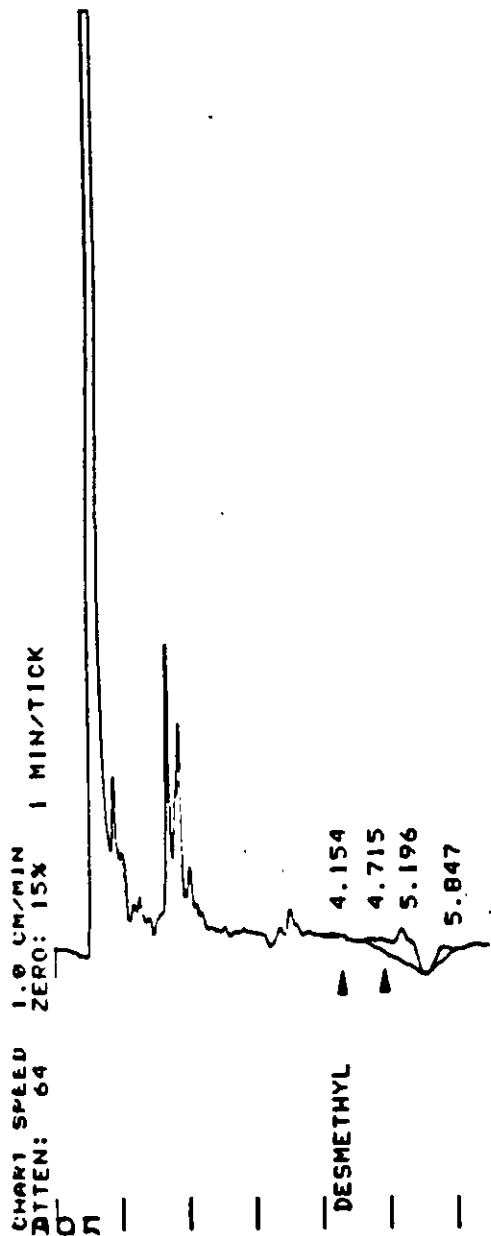
UNIDENT AREA: 2013

DETECTED PYS: 6 PEJECTED PKS: 1

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 27.9 OFFSET: 109

Figure 10. Representative EC-GC chromatogram of a whole orange check sample (01A-001); 1.0 mg equivalents injected; <0.01 ppm each of norflurazon and desmethyl norflurazon detected.



TITLE: NORF+DES IN CITRUS FRUITS 0:31 9 MAY 88

CHANNEL NO: 1 SAMPLE: CK LEM 46 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.0015	4.154	-0.096	193	BB	7.80
3	NORF	0.0000	4.804	0.014	722	VV	5.30
TOTALS:					0.0015	-0.082	915

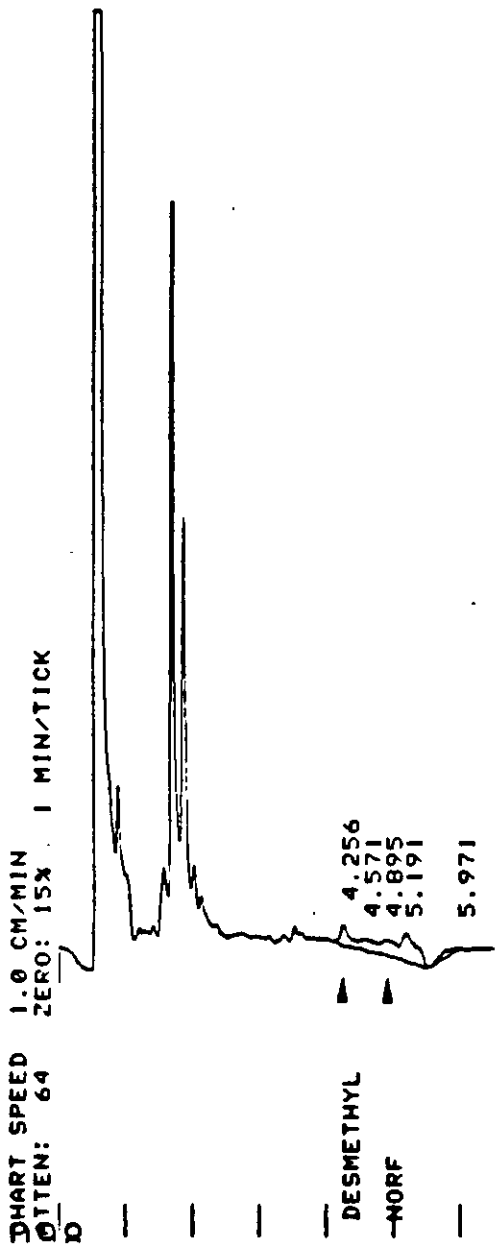
UNIDENT AREA: 2817

DETECTED PKS: 5 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 27.9 OFFSET: 111

Figure 11. Representative EC-GC chromatogram of a whole lemon check sample (86-01446); 1.0 mg equivalents injected; <0.01 ppm each of norflurazon and desmethyl norflurazon detected.



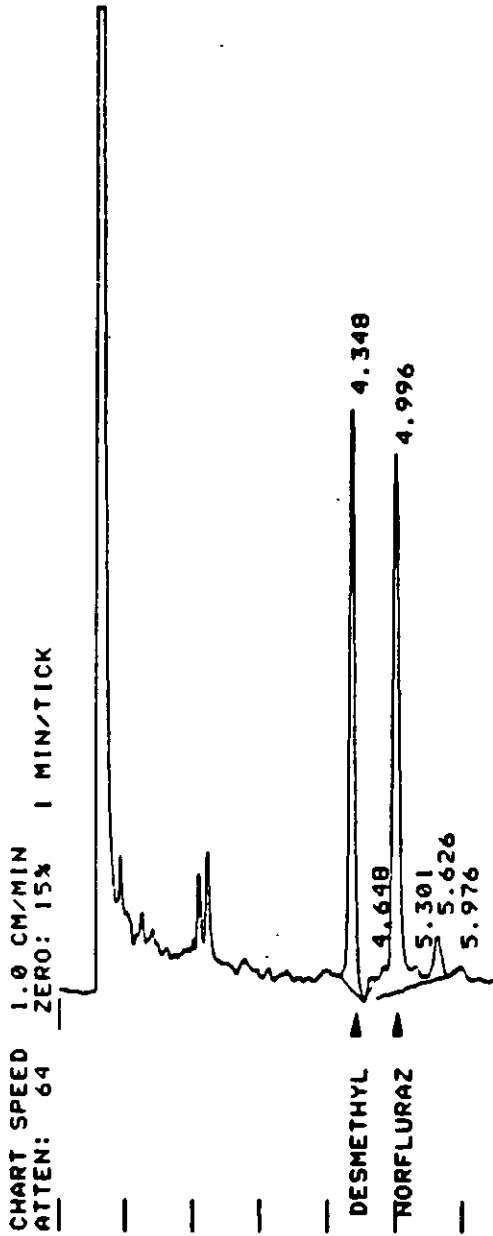
TITLE: NORF+DES IN CITRUS FRUITS 1:19 9 MAY 88
 CHANNEL NO: 1 SAMPLE: CK GR FR 50 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.0087	4.256	0.006	547.1086	BV	6.15
4	NORF	0.0173	4.895	0.015	224.924	VV	8.20
TOTALS:		0.0260		0.021	2010		

UNIDENT AREA: 2887
 DETECTED PKS: 6 REJECTED PKS: 1
 DIVISOR: 1.00000 MULTIPLIER: 1.00000
 NOISE: 27.9 OFFSET: 246

Figure 12. Representative EC-GC chromatogram of a whole grape fruit check sample (86-07050); 1.0 mg equivalents injected; <0.01 ppm each of norflurazon and desmethyl norflurazon detected.

145

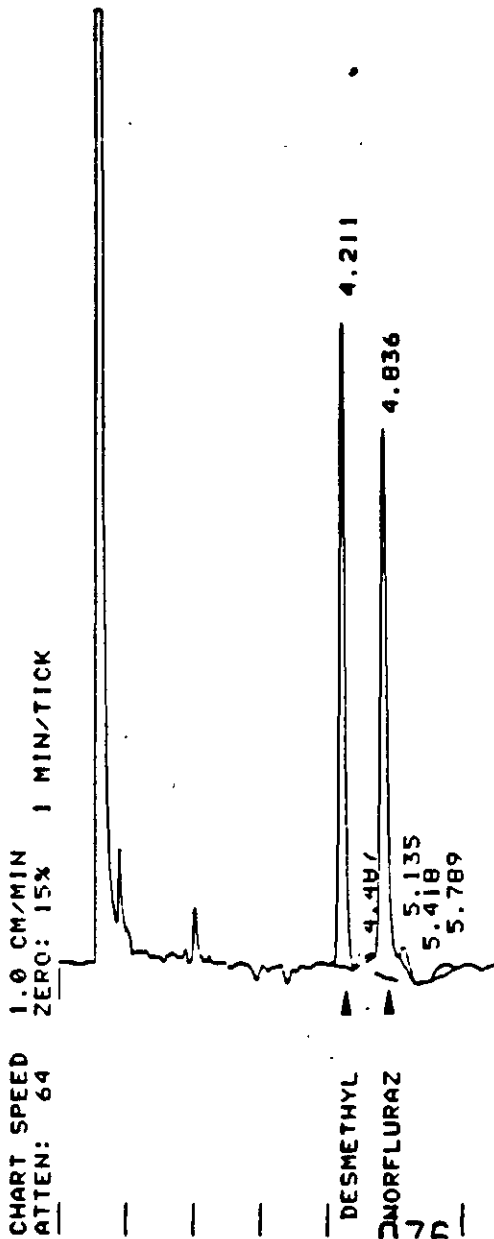


TITLE: NORF+DES IN ASPARAGUS 23:10 25 MAR 88

CHANNEL NO: 1 SAMPLE: 01427 CK+ 1 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.2665	4.348	-0.022	33273	BV	4.70
4	NORFLURAZ	0.3368	4.996	-0.024	30947	VV	5.85
TOTALS:		0.6033		-0.046	64220		
UNIDENT AREA:		6405					
DETECTED PKS:		0	REJECTED PKS:	0			
DIVISOR:		1.00000	MULTIPLIER:	1.00000			
NOISE:		35.2	OFFSET:	-641			

Figure 13. Representative EC-GC chromatogram of an asparagus check sample (86-01427); fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalents injected; 0.086 ng, 0.086 ppm norflurazon and 0.099 ng, 0.099 ppm desmethyl norflurazon detected. Recoveries were 86% for norflurazon and 99% for desmethyl norflurazon.



TITLE: NORF+DES IN PEANUTS 10:52 19 APR 88

CHANNEL NO: 1 SAMPLE: CK+MEAT#93 3 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.2973	4.211	-0.009	37126	BV	4.85
4	NORFLURAZ	0.3259	4.836	-0.004	34432	VV	5.90
TOTALS:					0.6232	-0.013	68558

UNIDENT AREA: 2519
 DETECTED PKS: 7 REJECTED PKS: 0
 DIVISOR: 1.00000 MULTIPLIER: 1.00000
 NOISE: 85.0 OFFSET: -68

Figure 14. Representative EC-GC chromatogram of a peanut meat check sample (86-06793) fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalents injected; 0.097 ng, 0.097 ppm norflurazon and 0.102 ng, 0.102 ppm desmethyl norflurazon detected. Recoveries were 97% for norflurazon and 102% for desmethyl norflurazon.

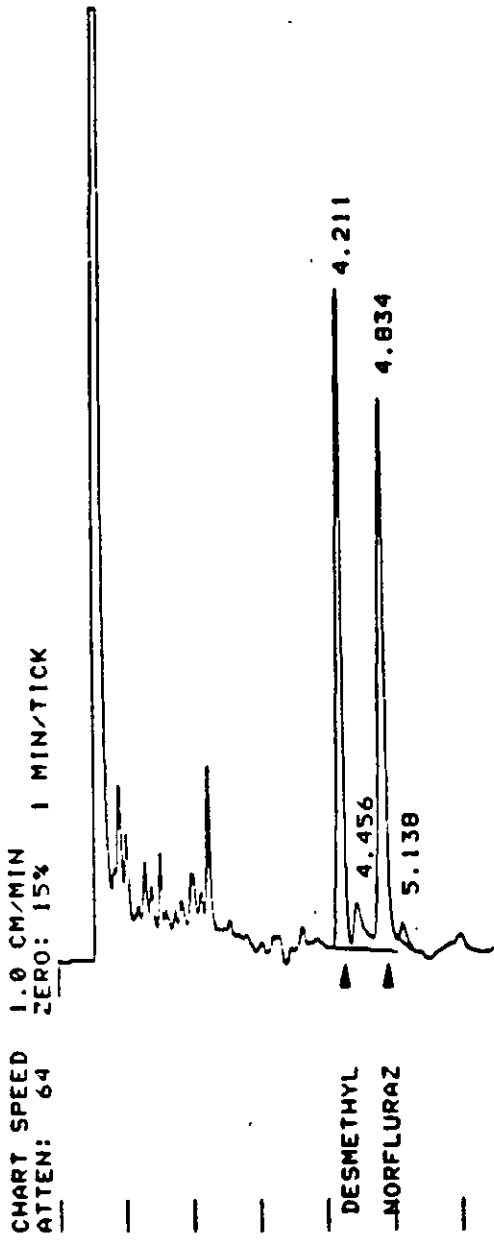


CHART SPEED 1.0 CM/MIN
ATTEN: 64 ZERO: 15% 1 MIN/TICK

DES METHYL 4.211
NOR FLURAZ 4.834

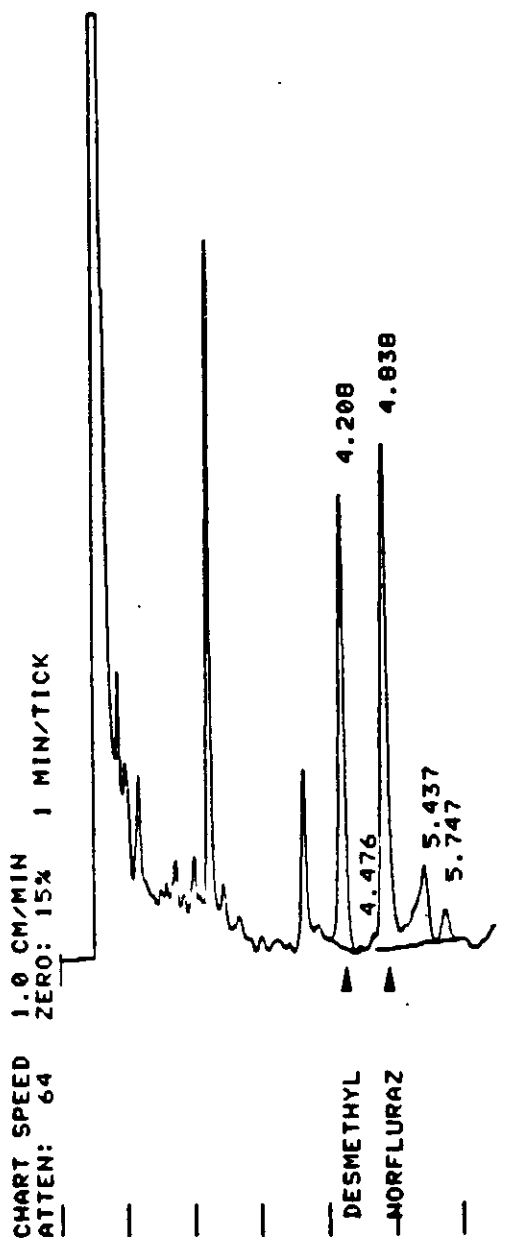
4.456
5.138

TITLE: NORF+DES IN PEANUTS 11:23 19 APR 88
CHANNEL NO: 1 SAMPLE: CK+HULL#93 1 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.3037	4.211	-0.009	364 3722	BV	4.80
3	NORFLURAZ	0.3289	4.834	-0.006	501 31722	VV	5.85
TOTALS:		0.6326		-0.015	69644		

UNIDENT AREA: 3716
DETECTED PKS: 4 REJECTED PKS: 0
DIVISOR: 1.00000 MULTIPLIER: 1.00000
NOISE: 85.0 OFFSET: -76

Figure 15. Representative EC-GC chromatogram of a peanut hull check sample (86-06793) fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalents injected; 0.098 ng, 0.098 ppm norflurazon and 0.10 ng, 0.10 ppm desmethyl norflurazon detected. Recoveries were 98% for norflurazon and 100% for desmethyl norflurazon.



TITLE: NORFLURAZES IN PEANUTS 14:16 21 APR 88

CHANNEL NO: 1 SAMPLE: CK+GRHAY 3 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.2020	4.208	-0.022	26038	BV	5.15
4	NORFLURAZ	0.3011	4.838	-0.022	29046	VV	6.20
TOTALS:		0.5031		-0.044	55084		

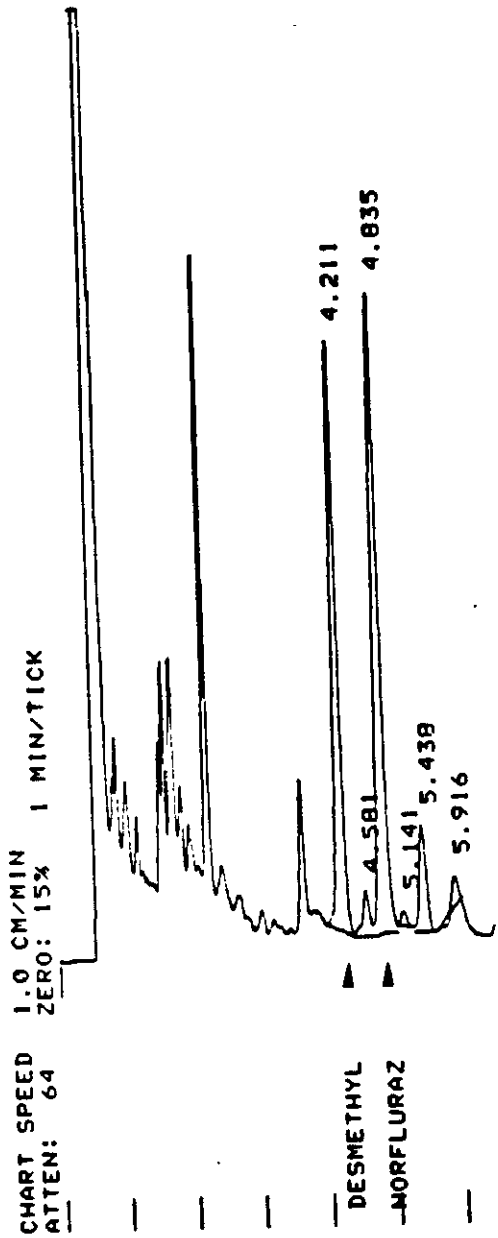
UNIDENT AREA: 6754

DETECTED PKS: 6 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 27.9 OFFSET: 26

Figure 16. Representative EC-GC chromatogram of a peanut green hay check sample (86-06784) fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalents injected; 0.095 ng, 0.095 ppm norflurazon and 0.083 ng, 0.083 ppm desmethyl norflurazon detected. Recoveries were 95% for norflurazon and 83% for desmethyl norflurazon.



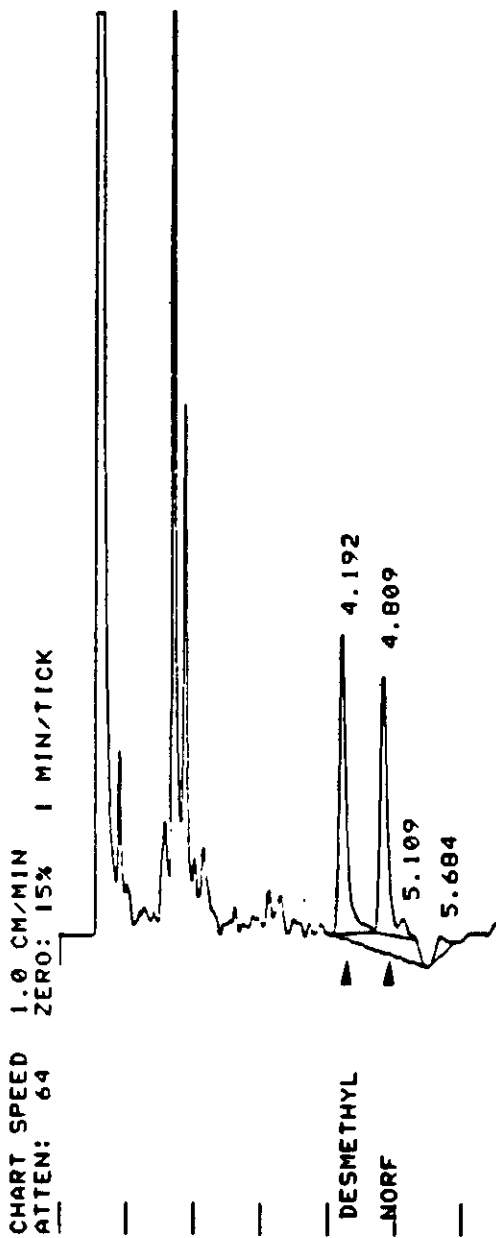
13:26 19 APR 88

TITLE: NORF+DES IN PEANUTS
CHANNEL NO: 1 SAMPLE: CK+DRHY#01 1 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	HI/2 (SEC)
1	DESMETHYL	0.2721	4.211	-0.009	37633970	BV	4.95
3	NORFLURAZ	0.3840	4.835	-0.005	3704230366	VV	5.90
TOTALS:							71020

UNIDENT AREA: 11028
DETECTED PKS: 6 REJECTED PKS: 0
DIVISOR: 1.00000 MULTIPLIER: 1.00000
NOISE: 85.0 OFFSET: -75

Figure 17. Representative EC-GC chromatogram of a peanut dry hay check sample (86-06801) fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalents injected; 0.099 ng, 0.099 ppm norflurazon and 0.09 ng, 0.09 ppm desmethyl norflurazon detected. Recoveries were 99% for norflurazon and 90% for desmethyl norflurazon.



TITLE: NORF+DES IN CITRUS FRUITS 17:13 10 MAY 88

CHANNEL NO: 1 SAMPLE: CK+S OR 001 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.1400	4.192	0.002	16309	BV	6.05
2	NORF	0.3049	4.809	-0.001	14721	VV	6.45

TOTALS: 0.4449 0.001 33370

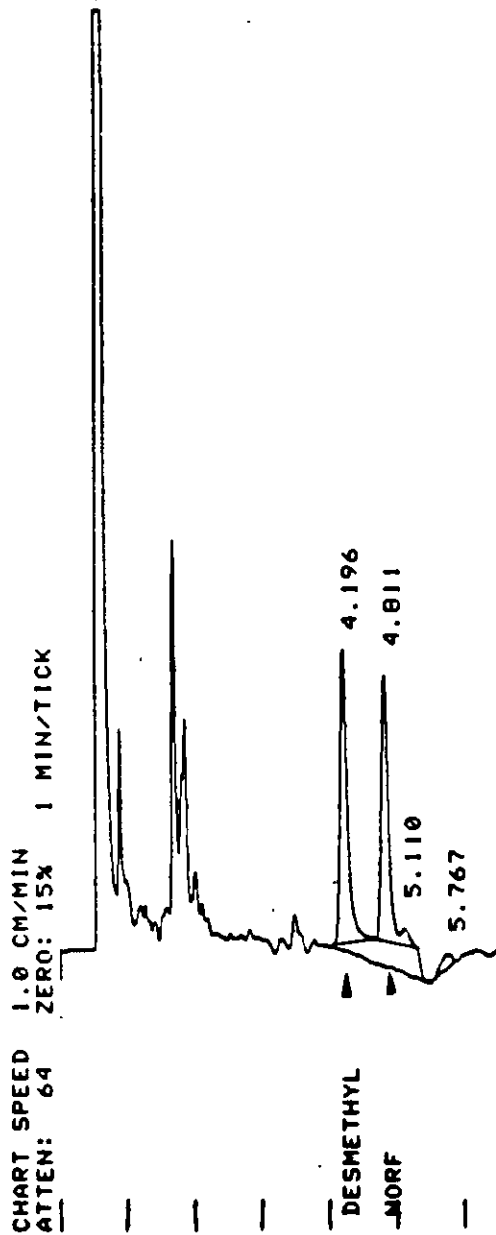
UNIDENT AREA: 3350

DETECTED PKS: 4 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 125.0 OFFSET: 460

Figure 18. Representative EC-GC chromatogram of a whole orange check sample (01A-001) fortified at 0.05 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalents injected; 0.049 ng, 0.049 ppm norflurazon and 0.05 ng, 0.05 ppm desmethyl norflurazon detected. Recoveries were 98% for norflurazon and 100% for desmethyl norflurazon.



TITLE: NORF+DES IN CITRUS FRUITS 18:26 10 MAY 88

CHANNEL NO: 1 SAMPLE: CK+2 LEM 46 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.1394	4.196	0.006	16125	BV	6.15
2	NORF	0.3213	4.811	0.001	15225	VV	6.50
TOTALS:		0.4607		0.007	34157		

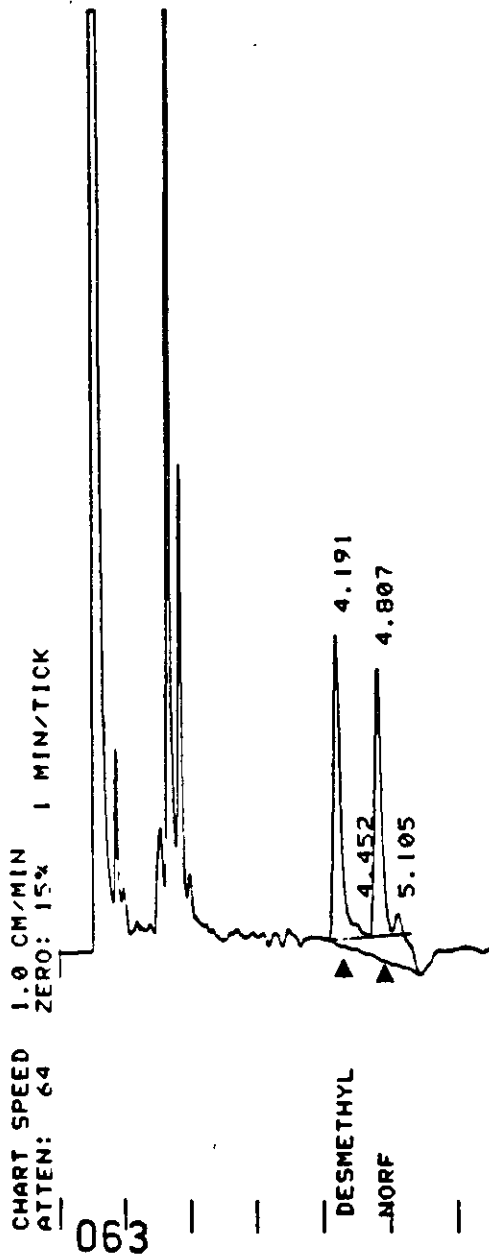
UNIDENT AREA: 3375

DETECTED PKS: 4 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 125.0 OFFSET: 112

Figure 19. Representative EC-GC chromatogram of a whole lemon check sample (86-01446) fortified at 0.05 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalents injected; 0.051 ng, 0.051 ppm norflurazon and 0.05 ng, 0.05 ppm desmethyl norflurazon detected. Recoveries were 102% for norflurazon and 100% for desmethyl norflurazon.



TITLE: NORF+DES IN CITRUS FRUITS 19:15 10 MAY 88

CHANNEL NO: 1 SAMPLE: CK+1 GRFR 50 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.1418	4.191	0.00117	224707	BV	6.10
3	NORF	0.3186	4.807	-0.00315	16607	VV	6.45

TOTALS: 0.4604 -0.002 34314

UNIDENT AREA: 4458

DETECTED PKS: 4 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

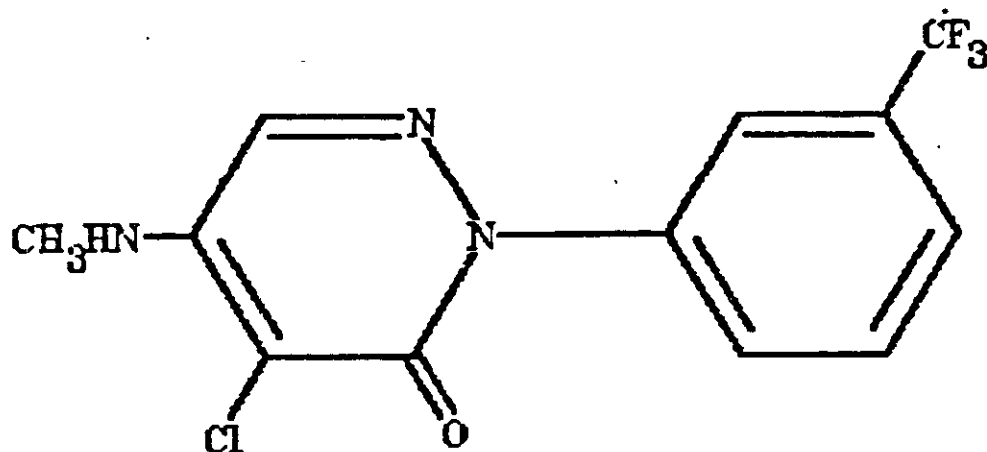
NOISE: 125.0 OFFSET: 63

Figure 20. Representative EC-GC chromatogram of a whole grape fruit check sample (86-07050) fortified at 0.05 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalents injected; 0.051 ng, 0.051 ppm norflurazon and 0.051 ng, 0.051 ppm desmethyl norflurazon detected. Recoveries were 102% for norflurazon and 102% for desmethyl norflurazon.

APPENDIX IV.

**REPRESENTATIVE STANDARD CURVES
AND CHROMATOGRAMS**

Norflurazon



desmethyl Norflurazon

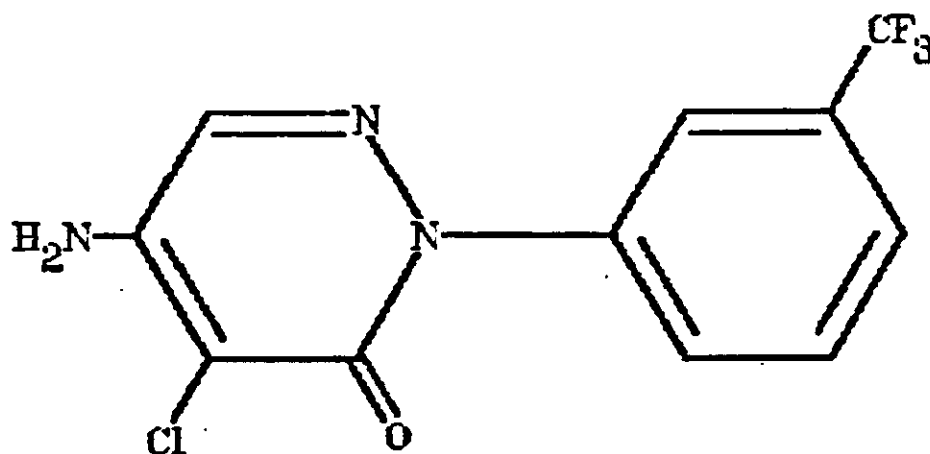


Figure 1. Chemical structures of norflurazon (4-chloro-5-(methylamino)-2-(α - α - α -trifluoro-m-tolyl)-3(2H)pyridazinone) and desmethyl norflurazon (5-amino-4-(chloro)-2-(α - α - α -trifluoro-m-tolyl)-3(2H)-pyridazinone).

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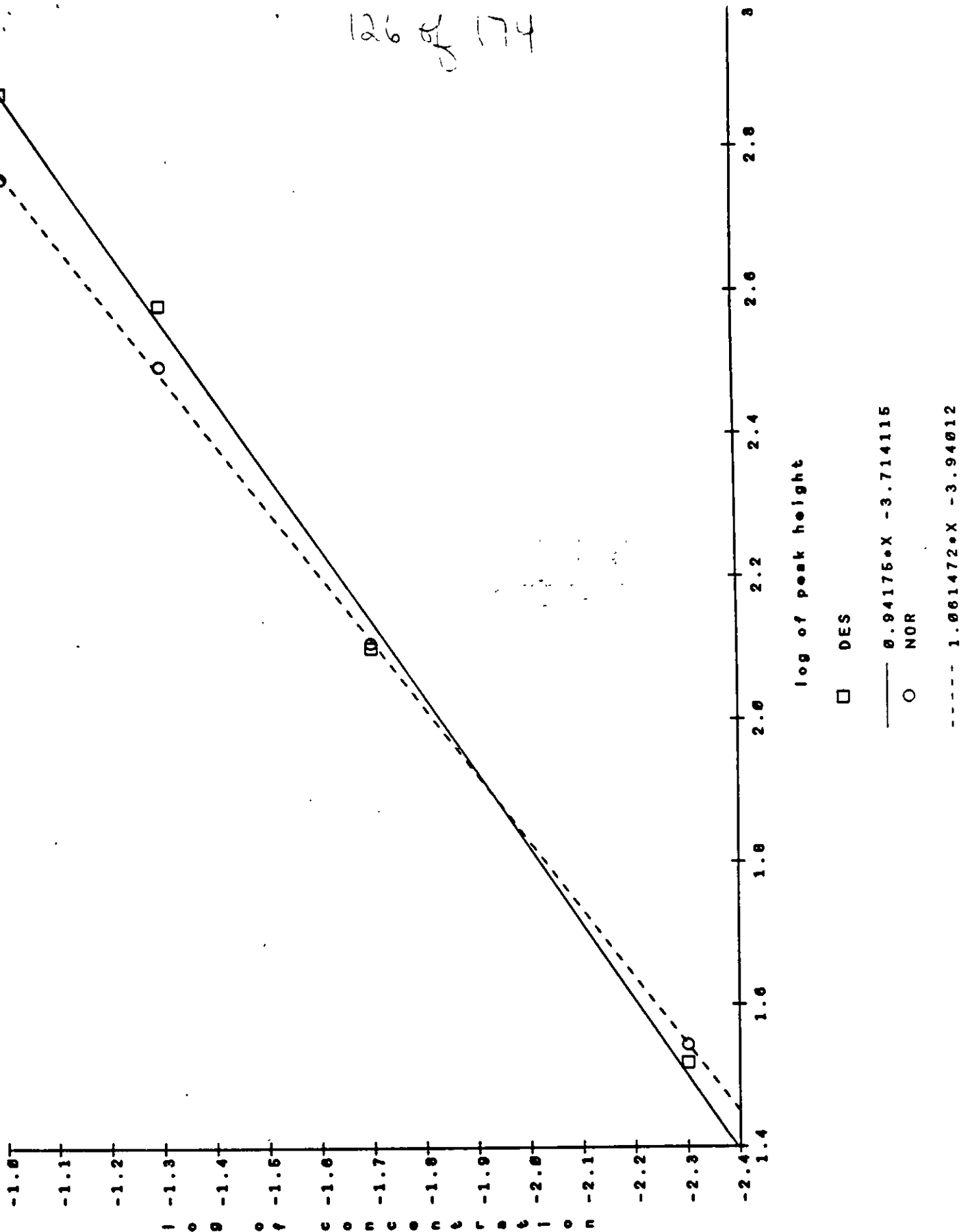
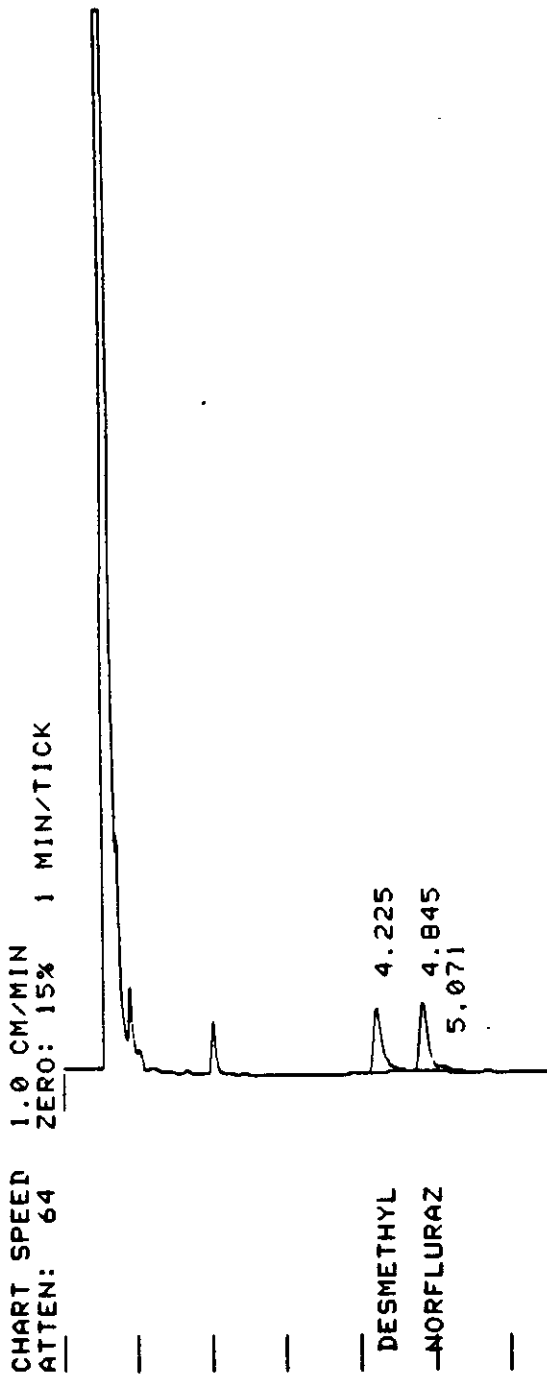


Figure 2. Representative computer generated standard curves of norflurazon and desmethyl norflurazon.

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TITLE: NORF+DES IN PEANUTS 11:08 19 APR 88

CHANNEL NO: 1 SAMPLE: 5X10-12 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT FACTOR	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.607533	4.225	0.005	3292	BB	6.70
2	NORFLURAZ	0.564972	4.845	0.005	3540	BV	6.55
TOTALS:					6832		

UNIDENT AREA: 231

DETECTED PKS: 3 REJECTED PKS: 0

AMT STD: 0.20000

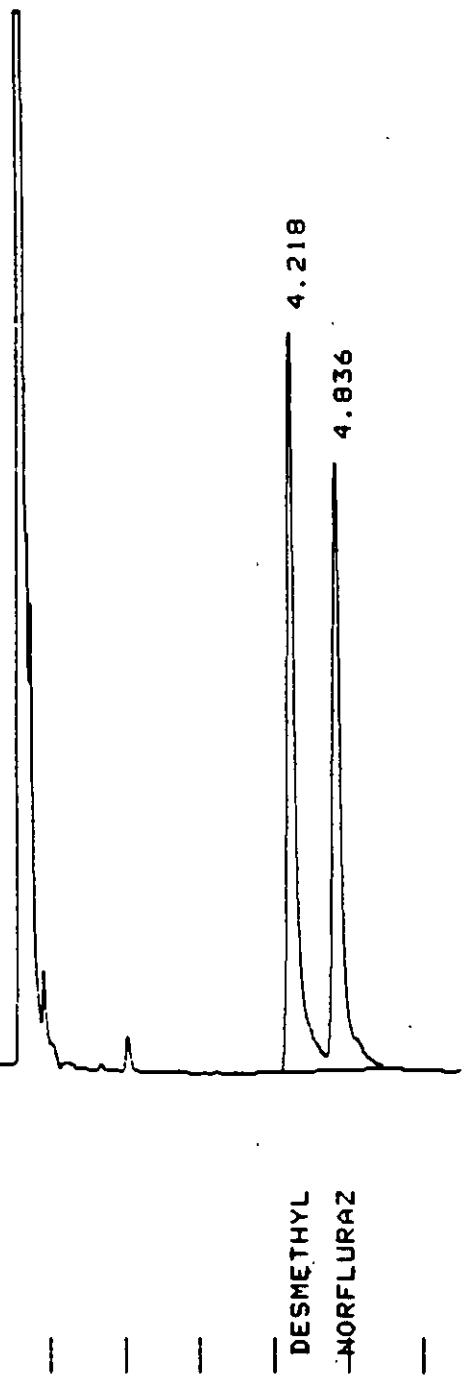
NOISE: 85.0 OFFSET: -54

ERRORS:
FACTOR NOT UPDATED

Figure 3. Representative EC-GC chromatogram of a 0.005 ng/uL each of norflurazon and desmethyl norflurazon GC standard; 2 uL injected.

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CHART SPEED 1.0 CM/MIN
ATTEN: 64 ZERO: 15% 1 MIN/TICK



TITLE: NORF+DES IN PEANUTS 13:11 19 APR 88

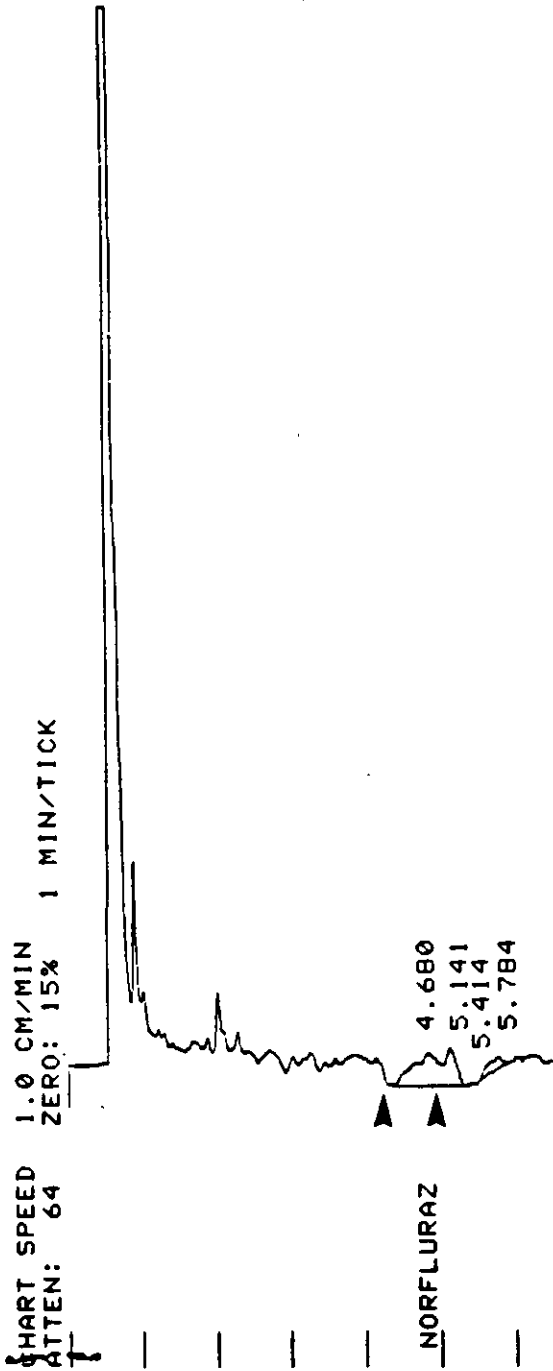
CHANNEL NO: 1 SAMPLE: 5X10-11 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT FACTOR	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.052217	4.218	-0.002	38302	BV	5.90
2	NORFLURAZ	0.063599	4.836	-0.004	31447	VB	6.50
TOTALS:					-0.006	69749	

UNIDENT AREA: 0
 DETECTED PKS: 2 REJECTED PKS: 0
 AMT STD: 0.20000
 NOISE: 85.0 OFFSET: 25
 ERRORS:
 FACTOR NOT UPDATED

Figure 4. Representative GC/EC chromatogram of a 0.05 ng/uL each of norflurazon and desmethyl norflurazon GC standard; 2 uL injected.

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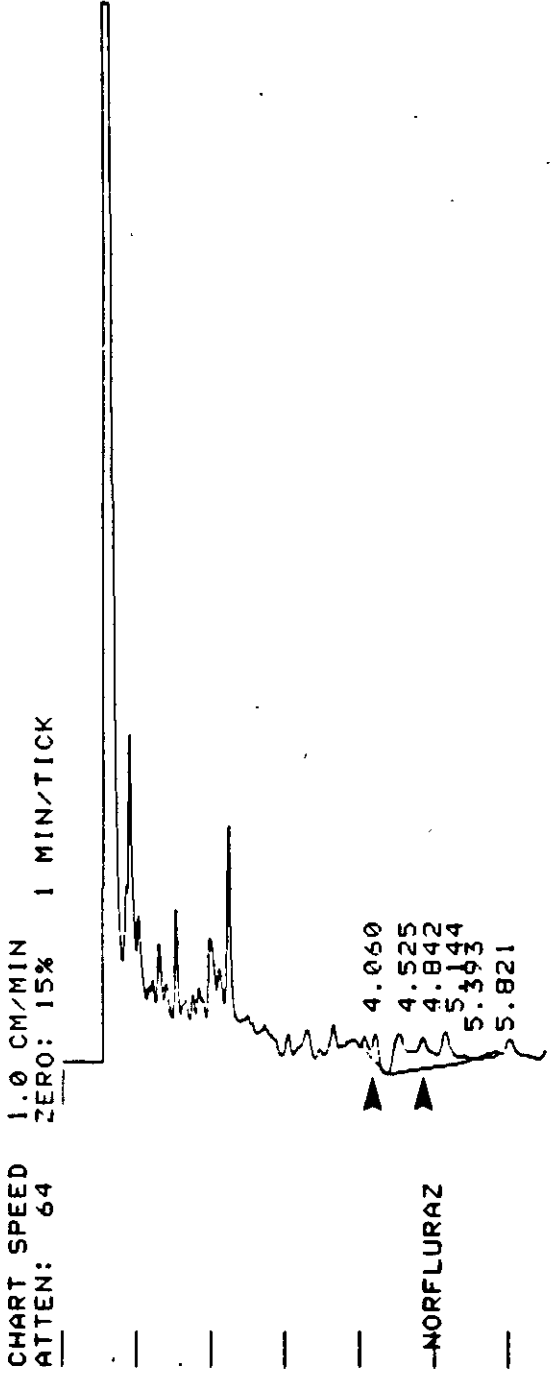
9:04 19 APR 88

TITLE: NORF+DES IN PEANUTS

CHANNEL NO: 1 SAMPLE: CK MEAT #93 METHOD: N+DI
 BEAK NO 2 NORFLURAZ RESULT 0.0175 TIME (MIN) 4.848
 TIME OFFSET 0.008 HEIGHT COUNTS 1686
 TOTALS: 0.0175 0.008 1686
 UNIDENT AREA: 3660
 DETECTED PKGS: 5 REJECTED PKGS: 1
 DIVISOR: 1.00000 MULTIPLIER: 1.00000
 NOISE: 85.0 OFFSET: -44

Figure 5. Representative EC-GC chromatogram of peanut meat check sample (86-06793); 1.0 mg equivalent injected; <0.01 ppm each of norflurazon and desmethyl norflurazon detected.

150 8/1/14



TITLE: NORF+DES IN PEANUTS 9:20 19 APR 88

CHANNEL NO: 1 SAMPLE: CK HULLS #93 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DESMETHYL	0.0118	4.206	-0.014	1473	VB	3.45
4	NORFLURAZ	0.0162	4.842	0.002	1560	VV	? 7.90
TOTALS:					0.0280	-0.012	3033

UNIDENT AREA: 4879

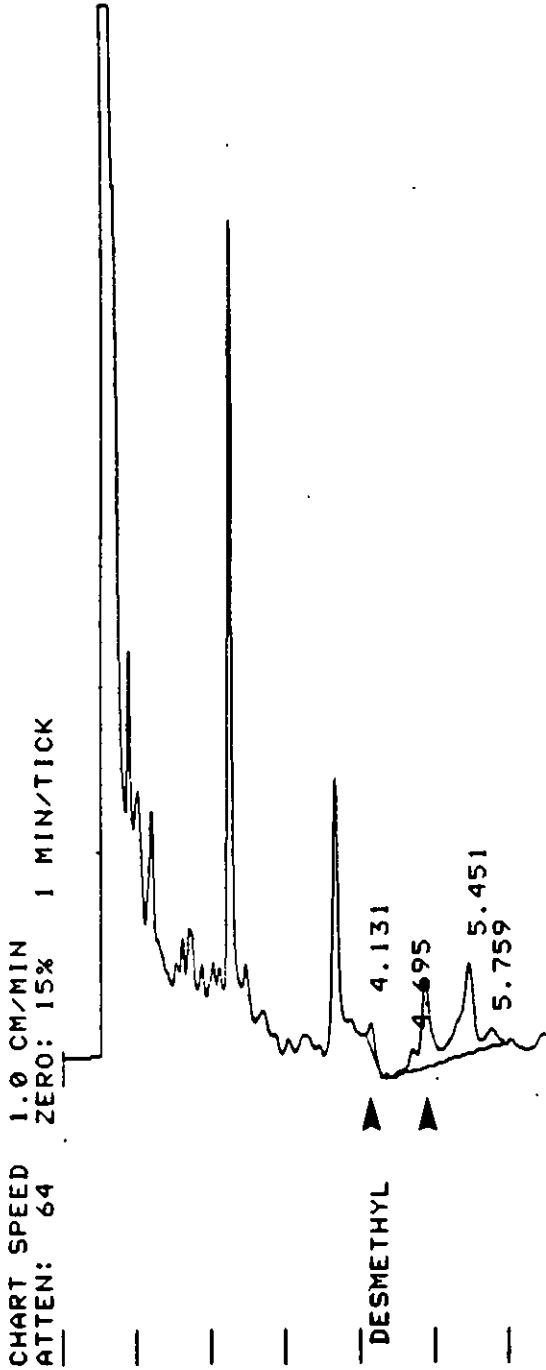
DETECTED PKS: 7 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 85.0 OFFSET: -21

Figure 6. Representative EC-GC chromatogram of peanut hulls check sample (86-06793); 1.0 mg equivalent injected; <0.01 ppm each of norflurazon and desmethyl norflurazon detected.

131 8/174



054

TITLE: NORF+DES IN PEANUTS 13:04 21 APR 88

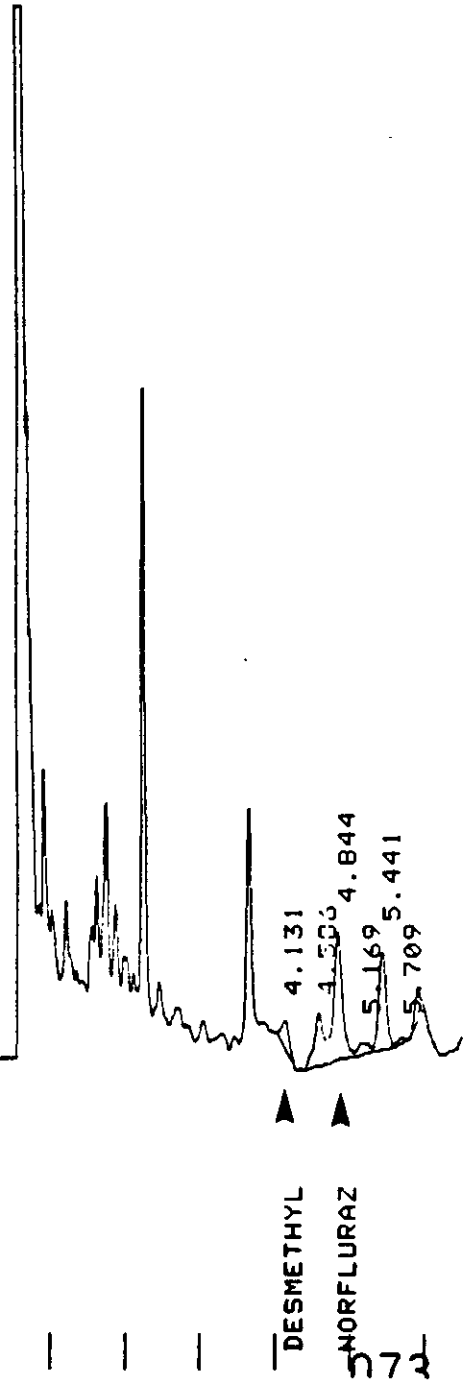
CHANNEL NO: 1 SAMPLE: CK GRHAY B4 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.0111	4.131	-0.099	1425	BB	3.70
4	NORFLURAZ	0.0468	4.859	-0.001	4512	VV	8.15
TOTALS:		0.0579		-0.100	5937		
UNIDENT AREA:		6885					
DETECTED PKS:		6	REJECTED PKS:		0		
DIVISOR:		1.00000	MULTIPLIER: 1.00000				
NOISE:		27.9	OFFSET: 19				

Figure 7. Representative EC-GC chromatogram of peanut green hay check sample (86-06784); 1.0 mg equivalent injected; <0.025 ppm of norflurazon and <0.01 ppm of desmethyl norflurazon detected.

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CHART SPEED 1.0 CM/MIN
ATTEN: 64 ZERO: 15% 1 MIN/TICK



TITLE: NORF+DES IN PEANUTS 9:50 19 APR 88

CHANNEL NO: 1 SAMPLE: CK DR HAY#01 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)	
1	DESMETHYL	0.0116	4.131	-0.089	1446	BB	3.90	
3	NORFLURAZ	0.0692	4.844	0.004	6674	VV	7.15	
TOTALS:					0.0808	-0.085	8120	

UNIDENT AREA: 10143

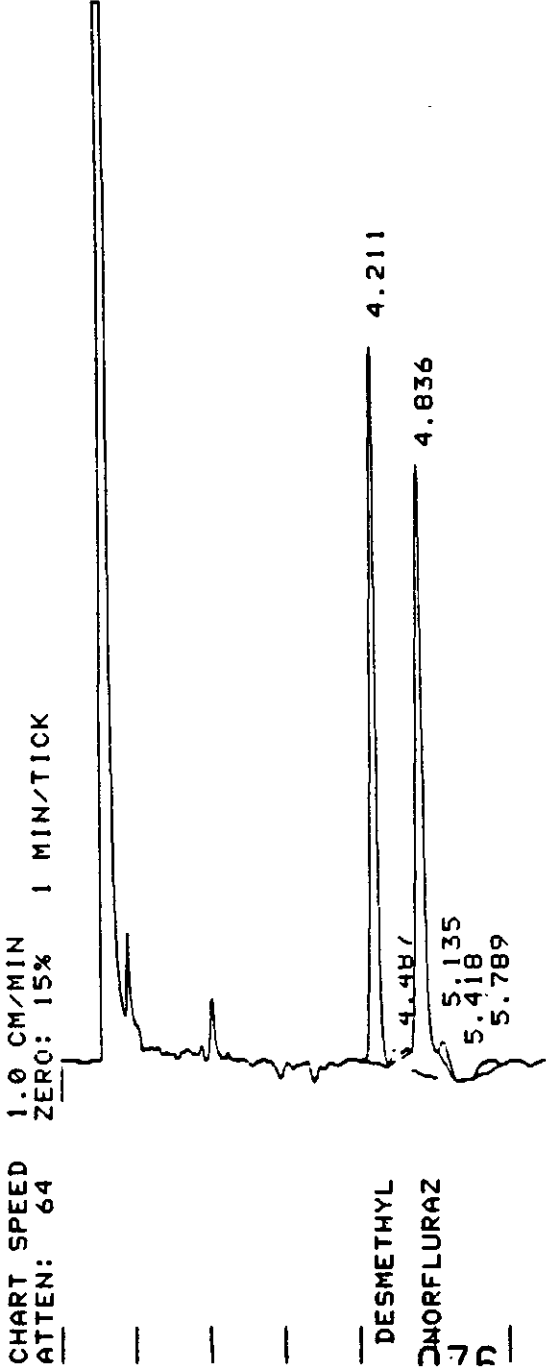
DETECTED PKGS: 8 REJECTED PKGS: 1

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 85.0 OFFSET: 65

Figure 8. Representative EC-GC chromatogram of peanut dry hay check sample (86-06801); 1.0 mg equivalent injected; <0.025 ppm of norflurazon and <0.01 ppm of desmethyl norflurazon detected.

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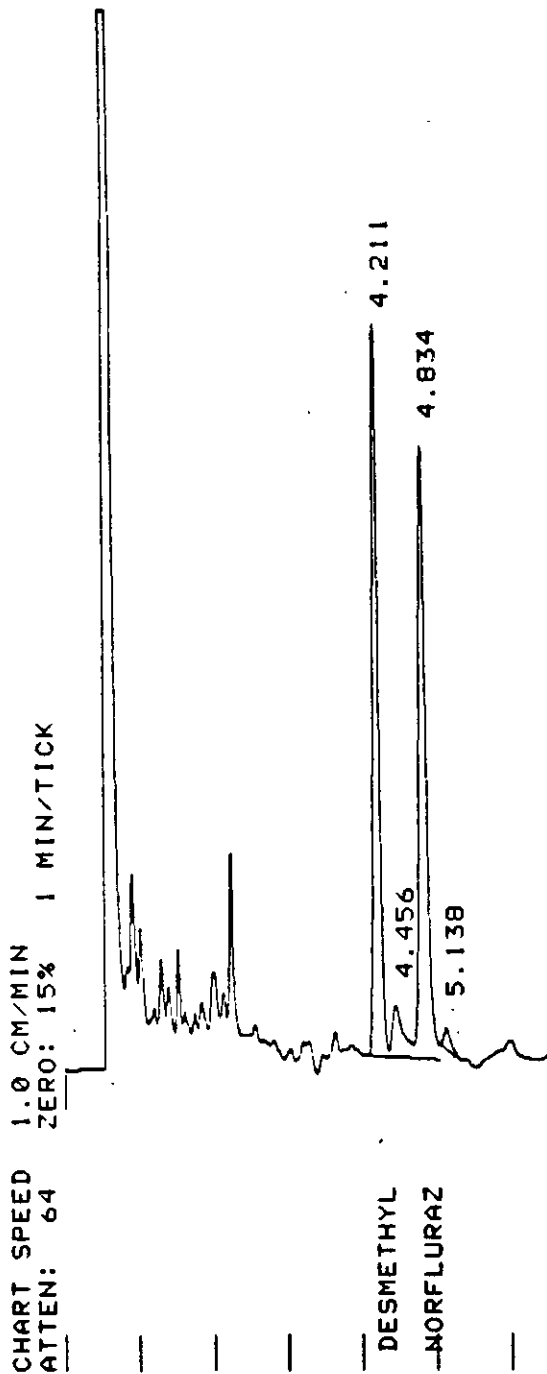
TITLE: NORF+DES IN PEANUTS 10:52 19 APR 88
CHANNEL NO: 1 SAMPLE: CK+MEAT#93 3. METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.2973	4.211	-0.009	37126	BV	4.85
4	NORFLURAZ	0.3259	4.836	-0.004	31432	VV	5.90
TOTALS:					0.6232	-0.013	68558

UNIDENT AREA: 2519
DETECTED PKGS: 7 REJECTED PKGS: 0
DIVISOR: 1.00000 MULTIPLIER: 1.00000
NOISE: 85.0 OFFSET: -68

Figure 9. Representative EC-GC chromatogram of peanut meat check sample (86-06793) fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalent injected; recoveries were 97.0% norflurazon and 102% desmethyl norflurazon.

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TITLE: NORF+DES IN PEANUTS 11:23 19 APR 88

CHANNEL NO: 1 SAMPLE: CK+HULL#93 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.3037	4.211	-0.009	364 37922	BV	4.80
3	NORFLURAZ	0.3289	4.834	-0.006	301 31722	VV	5.85

TOTALS: 0.6326 -0.015 69644

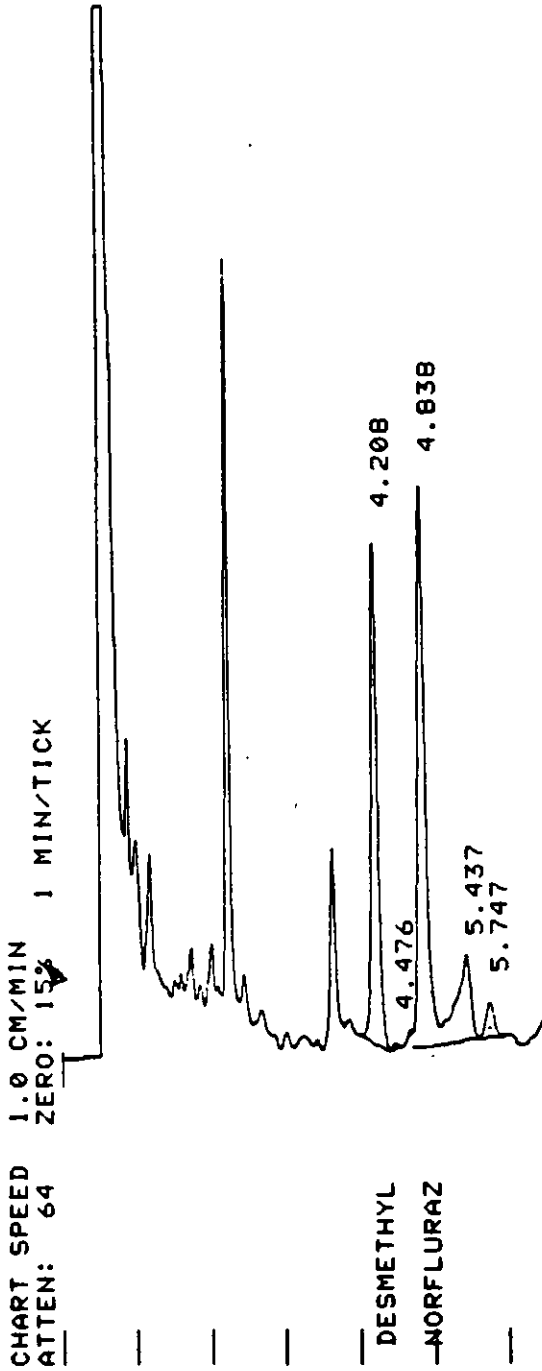
UNIDENT AREA: 3716

DETECTED PKS: 4 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 85.0 OFFSET: -76

Figure 10. Representative EC-GC chromatogram of peanut hulls check sample (86-06793) fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalent injected; recoveries were 98.0% norflurazon and 100% desmethyl norflurazon.



TITLE: NORF+DES IN PEANUTS 14:16 21 APR 88

CHANNEL NO: 1 SAMPLE: CK+GRHAY 3 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.2020	4.208	-0.022	26038	BV	5.15
4	NORFLURAZ	0.3011	4.838	-0.022	29046	VV	6.20
TOTALS:					0.5031	-0.044	55084

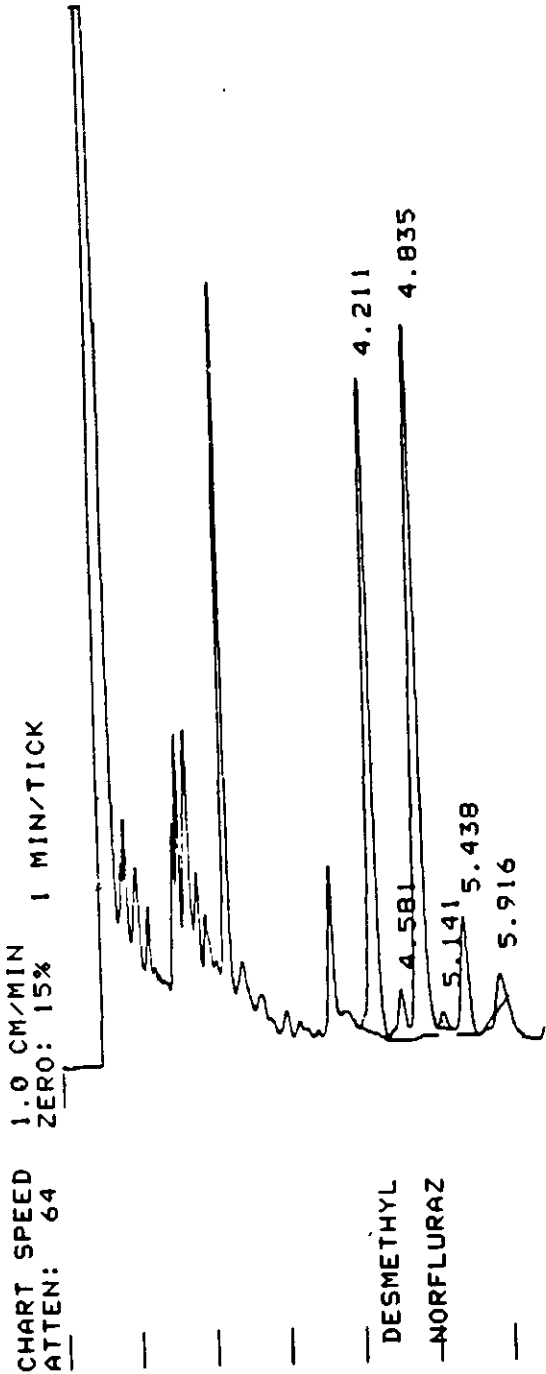
UNIDENT AREA: 6754

DETECTED PKS: 6 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 27.9 OFFSET: 26

Figure 11. Representative EC-GC chromatogram of peanut green hay check sample (86-06784) fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalents was injected; recoveries were 95.0% norflurazon and 83.0% desmethyl norflurazon.



13:26 19 APR 88

TITLE: NORF+DES IN PEANUTS

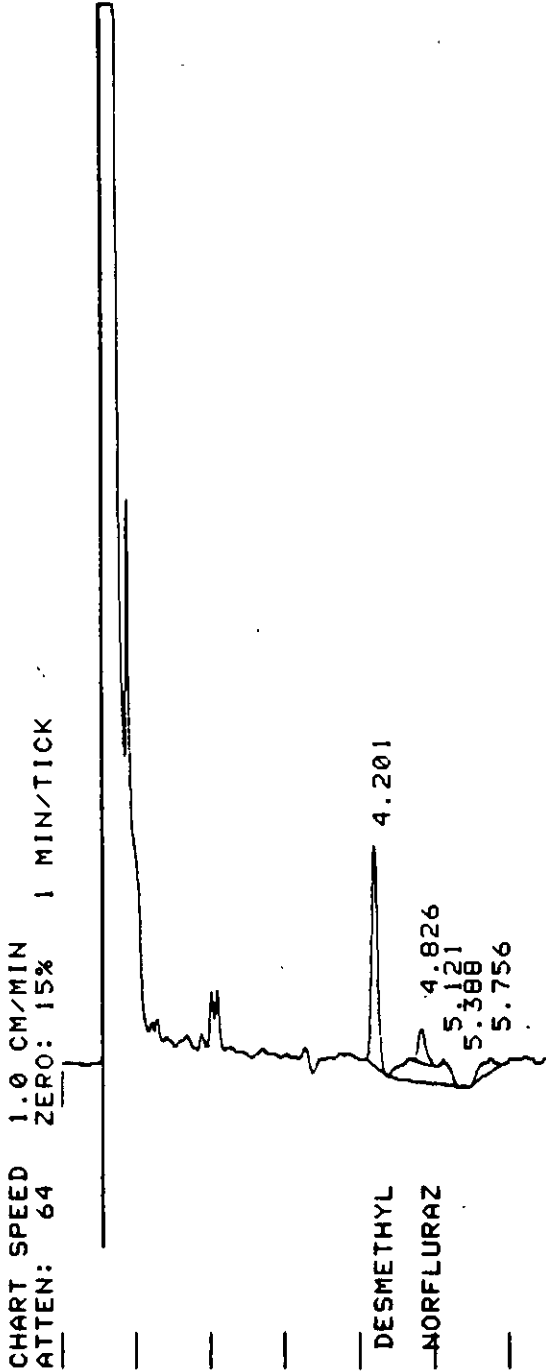
CHANNEL NO: 1 SAMPLE: CK+DRHY#01-1 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL NORFLURAZ	0.2721	4.211	-0.009	32633978	BV	4.95
3	NORFLURAZ	0.3840	4.835	-0.005	37042	VV	5.90
TOTALS:		0.6561		-0.014	71020		

UNIDENT AREA: 11028
 DETECTED PKS: 6 REJECTED PKS: 0
 DIVISOR: 1.00000 MULTIPLIER: 1.00000
 NOISE: 85.0 OFFSET: -75

Figure 12. Representative EC-GC chromatogram of peanut dry hay check sample (86-06801) fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon; 1.0 mg equivalent was injected; recoveries were 99.0% norflurazon and 90.0% desmethyl norflurazon.

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TITLE: NORF+DES IN PEANUTS 22:39 19 APR 88

CHANNEL NO: 1 SAMPLE: TR MEAT 96 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)	
1	DESMETHYL	0.0925	4.201	-0.019	11545	BV	4.90	
2	NORFLURAZ	0.0289	4.826	-0.014	2790	VV	8.00	
TOTALS:					0.1214	-0.033	14335	

UNIDENT AREA: 1781

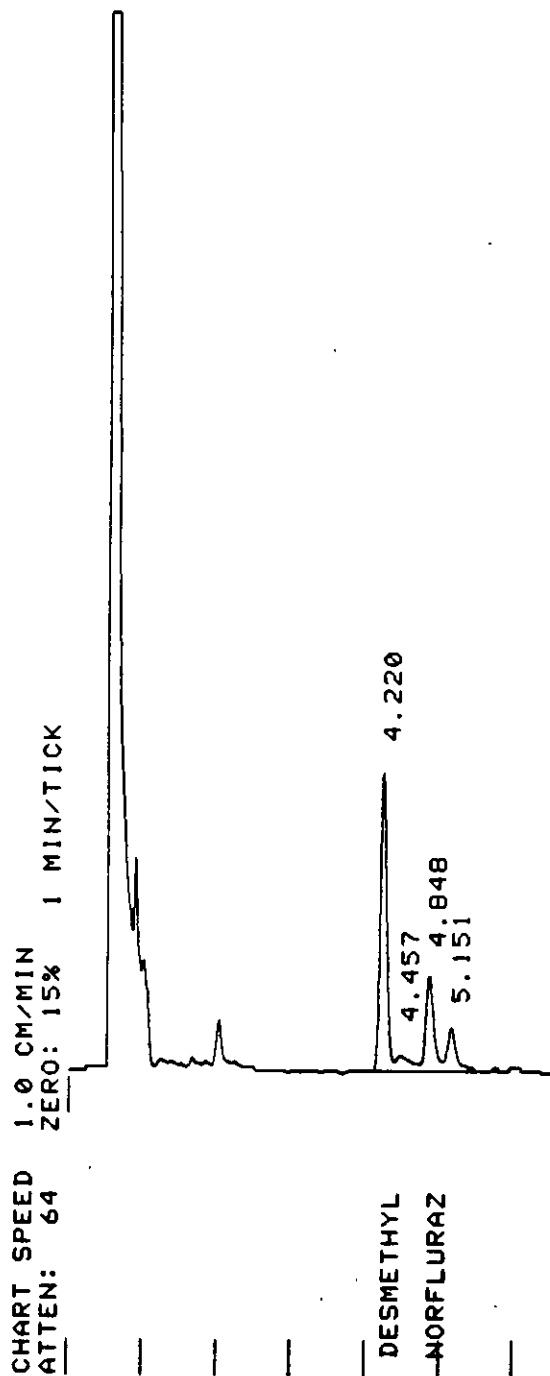
DETECTED PKS: 5 REJECTED PKS: 1

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 49.8 OFFSET: -29

Figure 13. Representative EC-GC chromatogram of peanut meat treatment sample (86-06796); 1.0 mg equivalent was injected; <0.01 ppm norflurazon and 0.032 ppm desmethyl norflurazon detected.

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15:14 21 APR 88

TITLE: NORF+DES IN PEANUTS

CHANNEL NO: 1 SAMPLE: TR HULLS 96 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DESMETHYL	0.1198	4.220	-0.010	15446	BV	5.55
3	NORFLURAZ	0.0509	4.848	-0.012	4910	VV	6.65
TOTALS:					0.1707	-0.022	20356

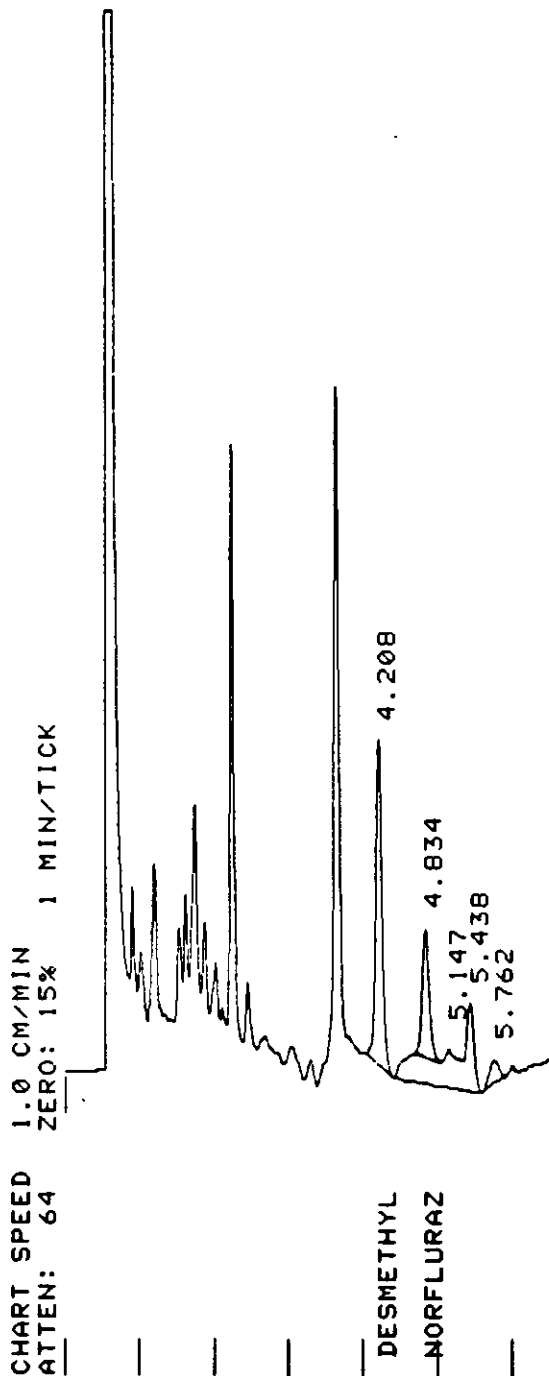
UNIDENT AREA: 3025

DETECTED PKS: 4 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 27.9 OFFSET: -12

Figure 14. Representative EC-GC chromatogram of peanut hulls treatment sample (86-06796); 0.066 mg equivalent was injected; 0.25 ppm norflurazon and 0.74 desmethyl norflurazon detected.



TITLE: NORF+DES IN PEANUTS 19:19 19 APR 88

CHANNEL NO: 1 SAMPLE: TR GRHAY B6 METHOD: N+DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	WI/2 (SEC)
1	DESMETHYL	0.1347	4.208	-0.012	16826	BV	4.90
2	NORFLURAZ	0.0823	4.834	-0.006	7934 6713	VV	6.70
TOTALS:		0.2170		-0.018	24760		

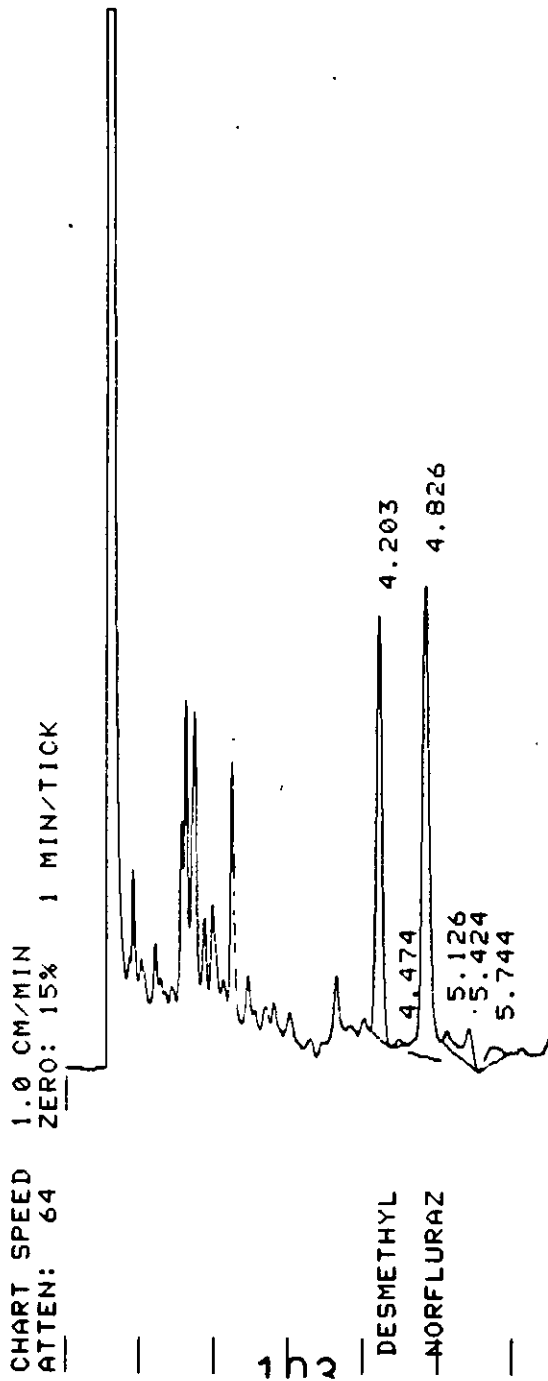
UNIDENT AREA: 7631

DETECTED PKS: 5 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIED: 1.00000

NOISE: 49.8 OFFSET: -47

Figure 15. Representative EC-GC chromatogram of peanut green hay treatment sample (86-06786); 1.0 mg equivalent injected 0.019 ppm norflurazon and 0.046 ppm desmethyl norflurazon detected.



20:51 19 APR 88

TITLE: NORF+DES IN PEANUTS

CHANNEL NO: 1 SAMPLE: TR DRHAY 114 METHOD: N+D1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	WI/2 (SEC)
1	DESMETHYL	0.1752	4.203	-0.017	21879	BV	4.75
3	NORFLURAZ	0.2530	4.826	-0.014	24406	VV	5.90

TOTALS: 0.4282 -0.031 46285

UNIDENT AREA: 3699

DETECTED PKS: 6 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 49.8 OFFSET: -28

Figure 16. Representative EC-GC chromatogram of peanut dry hay treatment sample (114); 1.0 mg equivalent was injected; 0.077 ppm norflurazon and 0.06 ppm desmethyl norflurazon detected.

Table 1: Recoveries of Norflurazon and Desmethyl Norflurazon from Check Peanut Meat, Hulls, and Hay Samples Fortified at 0.1 ppm each Compound.

Sample Type	Log No.	Replication	Percent Recovery	
			Norflurazon	Desmethyl Norflurazon
Nut meat	86-06793	1	106	93
		2	95	84
		3	97	102
		$\bar{x} \pm \text{sd}$	99.3 ± 5.9	93.0 ± 9.0
Hulls	86-06793	1	98	100
		2	101	104
		3	104	108
		$\bar{x} \pm \text{sd}$	101.0 ± 3.0	104 ± 4.0
Green hay	86-06784	1	92	80
		2	114	98
		3	95	83
		$\bar{x} \pm \text{sd}$	100.3 ± 11.9	87.0 ± 9.6
Dry hay	86-06801	1	99	90
		2	97	85
		3	81	87
		$\bar{x} \pm \text{sd}$	92.3 ± 9.9	87.3 ± 2.5