DETERMINATION OF NORFLURAZON AND ITS 
DESMETHYL METABOLITE IN RAW AGRICULTURAL COMMODITIES

1. SUMMARY

1.1 Scope

This method has been developed for the determination of norflurazon and its desmethyl metabolite (see figure 1 for structures) in raw agricultural commodities (RAC) for the purpose of determining residue tolerance levels and subsequently monitoring foodstuffs. This method was developed by the residue chemistry section of Sandoz Crop Protection Corporation.

The method is applicable for the determination of residues of norflurazon and its desmethyl metabolite in asparagus, lemon, grapefruit, orange, peanut hay (dry and green), peanut meat, and peanut hull with a limit of detection of 0.025 ppm for norflurazon in peanut hay (dry and green) and 0.01 ppm for norflurazon in all other matrices as well as 0.01 ppm for desmethyl norflurazon in all matrices tested.

1.2 Principle

1.2.1 A ten gram sample is blended in 70-100 mL of aqueous 0.5 N KOH and hydrolyzed for 1 hour at 90°C.

1.2.2 The hydrolysate is cooled to room temperature, 100 mL of methanol added, and the mixture shaken for 15 minutes.

1.2.3 The sample is then suction filtered, the filtrate adjusted to 200 mL with methanol and an aliquot representing 1 gram of substrate removed for cleanup.
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 × 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.
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 Funnel, 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. A16000.
3.1.20 Evaporator, N-Evap with 40°C water bath - (Organonation 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 COS 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.
3.2.2 Toluene, Baker Resi-Analyzed, J.T. Baker Company, Phillipsburg, 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-toly1)-3(2H)-pyridazinone and desmethyl norflurazon: 4-chloro-5-amino-2-(α,α,α-trifluoro-m-toly1)-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 x 10^{-8} g/μL for each compound.

3.3.3 Transfer 1.0 mL of the 1 x 10^{-6} g/μL stock solution from 3.3.2 to a 100-mL volumetric flask and bring to the mark with methanol. This solution (1 x 10^{-8} g/μL) 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 x 10^{-8} g/μL fortification standard from 3.3.3 to 100 mL (in a 100-mL volumetric flask) with toluene as follows:

<table>
<thead>
<tr>
<th>Volume of 1 x 10^{-8} g/μL standard solution</th>
<th>Concentration of final solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0 mL</td>
<td>1.0 x 10^{-10} g/μL</td>
</tr>
<tr>
<td>0.5 mL</td>
<td>5.0 x 10^{-11} g/μL</td>
</tr>
<tr>
<td>0.20 mL</td>
<td>2.0 x 10^{-11} g/μL</td>
</tr>
<tr>
<td>0.05 mL</td>
<td>5.0 x 10^{-12} g/μL</td>
</tr>
<tr>
<td>0.025 mL</td>
<td>2.5 x 10^{-12} g/μL</td>
</tr>
</tbody>
</table>
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 x 10^-8 g/µL 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.
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
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.
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:

\[
\text{PPM (ng/mg)} = \frac{C(\text{ng/µL}) \times V(\text{µL})}{W(\text{mg})}
\]

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
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 (X ± sd) for norflurazon ranged from 92.3 % ± 9.9 % (peanut green hay) to 102 % ± 4.0 % (grape fruit). Average recoveries for desmethyl norflurazon ranged from 87.0 % ± 9.6 % (peanut green hay) to 104.0 % ± 4.0 % (peanut hulls).
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.
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.

**NAME**

SYED ALI

**SIGNATURE**

[Signature]

**TITLE**

SENIOR SCIENTIST

**DATE**

6/10/88

---

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.

**NAME**

THOMAS BADE

**SIGNATURE**

[Signature]

**TITLE**

SENIOR SCIENTIST

**DATE**

6/10/88
Table 1. Recoveries Of Norflurazon And Desmethyl Norflurazon From Fortified Check Samples Of Various Raw Agricultural Commodities (RAC).

<table>
<thead>
<tr>
<th>RAC</th>
<th>Level of Fortification</th>
<th>Replication</th>
<th>Percent Recoveries</th>
</tr>
</thead>
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<tr>
<td></td>
<td></td>
<td></td>
<td>Desmethyl</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Norflurazon</td>
</tr>
<tr>
<td>1. Asparagus</td>
<td>0.1</td>
<td>1</td>
<td>86</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>2</td>
<td>98</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>3</td>
<td>94</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>4</td>
<td>80</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>5</td>
<td>87</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>6</td>
<td>84</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>$\bar{x} \pm s.d.$</td>
</tr>
<tr>
<td>2. Grapefruit</td>
<td>0.05</td>
<td>1</td>
<td>102</td>
</tr>
<tr>
<td></td>
<td>0.05</td>
<td>2</td>
<td>110</td>
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<td>3</td>
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</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>$\bar{x} \pm s.d.$</td>
</tr>
<tr>
<td>3. Lemon</td>
<td>0.05</td>
<td>1</td>
<td>78</td>
</tr>
<tr>
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<td>0.05</td>
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<td>100</td>
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<tr>
<td></td>
<td>0.05</td>
<td>3</td>
<td>94</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>$\bar{x} \pm s.d.$</td>
</tr>
<tr>
<td>4. Orange</td>
<td>0.05</td>
<td>1</td>
<td>96</td>
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<td></td>
<td>$\bar{x} \pm s.d.$</td>
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<td>5. Peanut</td>
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<td>1</td>
<td>80</td>
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<tr>
<td>green hay</td>
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<td>83</td>
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<td></td>
<td></td>
<td></td>
<td>$\bar{x} \pm s.d.$</td>
</tr>
<tr>
<td>6. Peanut</td>
<td>0.1</td>
<td>1</td>
<td>90</td>
</tr>
<tr>
<td>dry hay</td>
<td>0.1</td>
<td>2</td>
<td>85</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>3</td>
<td>87</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>$\bar{x} \pm s.d.$</td>
</tr>
<tr>
<td>7. Peanut</td>
<td>0.1</td>
<td>1</td>
<td>100</td>
</tr>
<tr>
<td>hull</td>
<td>0.1</td>
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<td>104</td>
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<td></td>
<td></td>
<td>$\bar{x} \pm s.d.$</td>
</tr>
<tr>
<td>8. Peanut</td>
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<td>1</td>
<td>93</td>
</tr>
<tr>
<td>meat</td>
<td>0.1</td>
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<tr>
<td></td>
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<td>3</td>
<td>102</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>$\bar{x} \pm s.d.$</td>
</tr>
</tbody>
</table>
Norflurazon

![Chemical structure of Norflurazon]

desmethyl Norflurazon

![Chemical structure of desmethyl Norflurazon]

Figure 1. Chemical Structures of Norflurazon: 4-chloro-5-(methylamino)-2-(α,α,α-trifluoro-m-toly)-3(2H)-pyridazinone and Desmethyl Norflurazon: 4-chloro-5-amino-2-(α,α,α-trifluoro-m-toly)-3(2H)-pyridazinone;
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.
Figure 3. Representative EC-EC chromatogram of 0.01 ng each of norflurazon and desmethyl norflurazon. A 2 µl injection of a 0.005 ng/µl standard solution.

<table>
<thead>
<tr>
<th>PEAK</th>
<th>NAME</th>
<th>RESULT</th>
<th>FACTOR</th>
<th>TIME</th>
<th>OFFSET</th>
<th>COUNTS</th>
<th>CODE</th>
<th>W1/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DESMETHYL</td>
<td>0.607533</td>
<td>4.225</td>
<td>0.005</td>
<td>3292</td>
<td>BB</td>
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<tr>
<td>2</td>
<td>NORFLURAZ</td>
<td>0.564972</td>
<td>4.845</td>
<td>0.005</td>
<td>3540</td>
<td>BV</td>
<td>6.85</td>
<td></td>
</tr>
</tbody>
</table>

TOTALS: 0.010  6832

UNIDENT AREA: 231
DETECTED PKS: 3  REJECTED PKS: 0

AMT STD: 0.20000
NOISE: 85.0  OFFSET: -54

ERRORS:
  FACTOR NOT UPDATED
Figure 4. Representative EC-GC chromatogram of 0.1 mg each of norflurazon and desmethyl norflurazon. A 2 µL injection of a 0.05 µg/µL standard solution.
Figure 5: Representative EC-CC chromatogram of an asparagus check sample (86-01427): 1.0 mg equivalents injected. Each of norflurazin and desmethylnorflurazin detected ppm.
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.
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.
Figure 8. Representative EC-GC chromatogram of peanut green hay check sample (66-06/84): 1.0 mg equivalents injected, 0.025 ppm norflurazon and < 0.01 ppm desmethyl norflurazon detected.

Title: Norf + Des in Peanuts

<table>
<thead>
<tr>
<th>Channel No: 1</th>
<th>Sample: CK GRHAY 84</th>
<th>Method: N+D1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peak No</td>
<td>Name</td>
<td>Result</td>
</tr>
<tr>
<td>1</td>
<td>Desmethyl</td>
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</tr>
<tr>
<td>4</td>
<td>Norfluraz</td>
<td>0.0468</td>
</tr>
</tbody>
</table>

Totals:

<table>
<thead>
<tr>
<th>Result</th>
<th>Offset</th>
<th>SEC</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0579</td>
<td>-0.100</td>
<td>5937</td>
</tr>
</tbody>
</table>

Unidentified Area: 6885

Detected PKs: 6
Rejected PKs: 0

Divisor: 1.00000
Multiplier: 1.00000

Noise: 27.9 Offset: 19
Figure 9: Representative GC chromatogram of a peanut dry hay check sample (86-0680): 1.0 mg equivalents injected.

<table>
<thead>
<tr>
<th>No.</th>
<th>Peak Name</th>
<th>Result 4.131</th>
<th>Time (Min)</th>
<th>Offset</th>
<th>Height</th>
<th>SEp</th>
<th>Code</th>
<th>W1/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Desmethyl</td>
<td>0.0116</td>
<td>4.131</td>
<td>-0.009</td>
<td>1446</td>
<td>BB</td>
<td>3.90</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Norfluraz</td>
<td>0.0692</td>
<td>4.844</td>
<td>0.004</td>
<td>6674</td>
<td>VV</td>
<td>7.15</td>
<td></td>
</tr>
</tbody>
</table>

**Totals:**

<table>
<thead>
<tr>
<th>Result</th>
<th>Offset</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0080</td>
<td>-0.005</td>
<td>8120</td>
</tr>
</tbody>
</table>

**Unident Area:** 10143

**Detected PKS:** 0  **Rejected PKS:** 1

**Divisor:** 1.00000  **Multiplier:** 1.00000

**Noise:** 85.0  **Offset:** 65
Figure 10. Representative GC chromatogram of orange juice sample, at 0.1 mg equivalents injected. 400 ppm each of norfuranon and desmethylnorfuranon detected.

<table>
<thead>
<tr>
<th>PEAK NO.</th>
<th>PEAK</th>
<th>RESULT</th>
<th>TIME (MIN)</th>
<th>OFFSET</th>
<th>COUNTS</th>
<th>CODE</th>
<th>WI/2 (SEC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DESMETHYL</td>
<td>0.0038</td>
<td>4.241</td>
<td>-0.009</td>
<td>479</td>
<td>001</td>
<td>7.10</td>
</tr>
<tr>
<td>3</td>
<td>NORF</td>
<td>0.0000</td>
<td>4.092</td>
<td>0.012</td>
<td>2139</td>
<td>001</td>
<td>7.10</td>
</tr>
</tbody>
</table>

TOTALS: 0.0038 0.003 2618

UNIDENT AREA: 2013
DETECTED PKS: 6  REJECTED PKS: 1
DIVISOR: 1.00000  MULTIPLIER: 1.00000
NOISE: 27.9  OFFSET: 109
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.
Figure 12: Representative GC/EC chromatogram of a whole grapefruit sample (86-07050); 1.0 mg equivalents injected. 40.01 ppm each of norflurazon and desmethyl norflurazon detected.

**TITLE:** NORF+DES IN CITRUS FRUITS  
**CHANNEL NO:** 1  
**SAMPLE:** CK GR FR 50  
**METHOD:** N+DI

<table>
<thead>
<tr>
<th>PEAK NUMBER</th>
<th>NAME</th>
<th>PEAK RESULT</th>
<th>TIME (MIN)</th>
<th>OFFSET</th>
<th>COUNTS</th>
<th>CODE</th>
<th>WI/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DESMETHYL</td>
<td>0.0087</td>
<td>4.256</td>
<td>0.006</td>
<td>547,1086</td>
<td>BV</td>
<td>6.15</td>
</tr>
<tr>
<td>4</td>
<td>NORF</td>
<td>0.0173</td>
<td>4.895</td>
<td>0.015</td>
<td>12V, 924</td>
<td>VV</td>
<td>8.20</td>
</tr>
</tbody>
</table>

**TOTALS:** 0.0260  
**UNIDENTIFIED AREA:** 2887

**DETECTED PKS:** 6  
**REJECTED PKS:** 1

**DIVISOR:** 1.00000  
**MULTIPLIER:** 1.00000

**NOISE:** 27.9  
**OFFSET:** 246
Figure 13. Representative GC chromatogram of an asparagus check sample (86-01427) fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon. Recoveries were 88% for norflurazon and 96% for desmethyl norflurazon.

<table>
<thead>
<tr>
<th>CHANNEL NO.</th>
<th>SAMPLE: 01427 CK+1</th>
<th>METHOD: N+D1</th>
</tr>
</thead>
<tbody>
<tr>
<td>PEAK NO.</td>
<td>RESULT</td>
<td>TIME</td>
</tr>
<tr>
<td>1</td>
<td>DESMETHYL</td>
<td>0.2665</td>
</tr>
<tr>
<td>4</td>
<td>NORFLURAZ</td>
<td>0.3338</td>
</tr>
<tr>
<td>TOTALS</td>
<td>0.6033</td>
<td>-0.046</td>
</tr>
</tbody>
</table>

UNIDENT AREA: 6405

DETECTED PKS: 0
REJECTED PKS: 0
DIVISOR: 1.00000
MULTIPLIER: 1.00000
NOISE: 35.2
OFFSET: -641
Figure 14. Representative GC-EC chromatogram of a peanut meat check sample (86-06793) fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon. Recoveries were 97% for norflurazon and 102% for desmethyl norflurazon.

**Title**: NOFF+DES IN PEANUTS

**Channel No.**: 1

**Sample**: CK+MEAT#93 3

**Method**: N+DI

<table>
<thead>
<tr>
<th>Peak Name</th>
<th>Result</th>
<th>Time (min)</th>
<th>Offset</th>
<th>Height</th>
<th>SEP</th>
<th>Code</th>
<th>W1/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Desmethyl</td>
<td>0.2973</td>
<td>4.211</td>
<td>-0.009</td>
<td>37126</td>
<td>BV</td>
<td>4.05</td>
<td></td>
</tr>
<tr>
<td>Norfluraz</td>
<td>0.3259</td>
<td>4.636</td>
<td>-0.004</td>
<td>31432</td>
<td>VV</td>
<td>5.90</td>
<td></td>
</tr>
</tbody>
</table>

| 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**: -60
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.
Figure 16. Representative GC-GC chromatogram of peanut green hay, chart speed 1.00 cm/min.

<table>
<thead>
<tr>
<th>NAME</th>
<th>RESULT (µg/mL)</th>
<th>TIME (MIN)</th>
<th>TIME (SEC)</th>
<th>HEIGHT</th>
<th>SEP</th>
<th>W1/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>DESMETHYL</td>
<td>0.2020</td>
<td>4.208</td>
<td>-0.022</td>
<td>26038</td>
<td>BV</td>
<td>5.15</td>
</tr>
<tr>
<td>NORFLURAZ</td>
<td>0.3011</td>
<td>4.838</td>
<td>-0.022</td>
<td>29046</td>
<td>VV</td>
<td>6.20</td>
</tr>
<tr>
<td>TOTALS:</td>
<td>0.5031</td>
<td>-0.044</td>
<td>55084</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

UNIDENTIFIED AREA: 6754

DETECTED PKS: 6
REJECTED PKS: 0

DIVIDER: 1.00000
MULTIPLIER: 1.00000

NOISE: 27.9
OFFSET: 26
Figure 17. Representative EC-GC chromatogram of a peanut dry hay check sample (66-0680) fortified at 0.1 ppm each of norflurazon and desmethylnorflurazon. 1.0 mg equivalents of norflurazon and 0.99 mg norflurazon were injected. 0.099 mg desmethylnorflurazon and 0.099 mg desmethylnorflurazon detected. Recoveries were 99% for norflurazon and 90% for desmethylnorflurazon.

**Chart Speed:** 1.0 cm/min  
**Attenuation:** 64  
**Zero:** 15%  
**1 Min/Tick**

**Title:** NORF+DES in PEANUTS  
**Sample:** CK+DRHY#01  
**Method:** N+D1

<table>
<thead>
<tr>
<th>PEAK NO</th>
<th>NAME</th>
<th>RESULT</th>
<th>TIME (MIN)</th>
<th>TIME OFFSET</th>
<th>COUNTS</th>
<th>CODE</th>
<th>W1/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DESMETHYL</td>
<td>0.2721</td>
<td>4.211</td>
<td>-0.009</td>
<td>371.33976</td>
<td>BV</td>
<td>4.95</td>
</tr>
<tr>
<td>3</td>
<td>NORFLURAZ</td>
<td>0.3840</td>
<td>4.835</td>
<td>-0.005</td>
<td>339.042</td>
<td>VV</td>
<td>5.90</td>
</tr>
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</table>

**Totals:** 0.6561  
**Unidentified Area:** 11.028

**Detected PKS:** 6  
**Rejected PKS:** 0

**Divisor:** 1.00000  
**Multiplier:** 1.00000

**Noise:** 85.0  
**Offset:** -75
Figure 10. Representative EC-GC chromatogram of a whole orange check sample (GLA-001) fortified at 0.05 ppm each of norflurazon and desmethylnorflurazon. Recoveries were 98% for norflurazon and 100% for desmethylnorflurazon.

Title: NORF+DES IN CITRUS FRUITS

Channel No: 1  Sample: CK+5 OR 001  Method: N+D1

<table>
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<th>Peak</th>
<th>Name</th>
<th>Result</th>
<th>Time (Min)</th>
<th>Offset</th>
<th>Height</th>
<th>SEP</th>
<th>Code (Sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Desmethylnorflurazon</td>
<td>0.1400</td>
<td>4.192</td>
<td>0.002</td>
<td>1419</td>
<td>BV</td>
<td>6.05</td>
</tr>
<tr>
<td>2</td>
<td>NORF</td>
<td>0.3049</td>
<td>4.809</td>
<td>-0.001</td>
<td>1502</td>
<td>VV</td>
<td>6.45</td>
</tr>
</tbody>
</table>

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 19. Representative EC-GL chromatogram of a whole lemon check sample (66-01446) fortified at 0.05 ppm each of norflurazon and desmethyl norflurazon: 1.0 ppm norflurazon detected. Recoveries were 102% for norflurazon and 100% for desmethyl norflurazon.

**Title:** NORF+DES IN CITRUS FRUITS  
**Channel No.:** 1  
**Sample:** CK+2 LEM 46  
**Method:** N+DI

<table>
<thead>
<tr>
<th>Peak No.</th>
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<th>Result (ng)</th>
<th>Time (min)</th>
<th>Time Offset</th>
<th>Counts</th>
<th>Code (sec)</th>
<th>$W_1/2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Desmethyl</td>
<td>0.1394</td>
<td>4.196</td>
<td>0.006</td>
<td>1615</td>
<td>41396</td>
<td>6.15</td>
</tr>
<tr>
<td>2</td>
<td>Norf</td>
<td>0.3213</td>
<td>4.811</td>
<td>0.001</td>
<td>15775</td>
<td>46748</td>
<td>6.50</td>
</tr>
<tr>
<td>Totals</td>
<td></td>
<td>0.4607</td>
<td></td>
<td>0.007</td>
<td></td>
<td>34157</td>
<td></td>
</tr>
</tbody>
</table>

**Unidentified Area:** 3375

**Detected Pks:** 4  
**Rejected Pks:** 0

**Divisor:** 1.00000  
**Multiplier:** 1.00000

**Noise:** 125.0  
**Offset:** 112
**Figure 20.** Representative EC-GC chromatogram of a whole grapefruit check sample (86-0705) fortified at 0.05 ppm each of norflurazon and desmethyl norflurazon; 1.6 ppm equivalents of norflurazon; 0.051 ppm desmethyl norflurazon; and 0.051 ppm norflurazon detected. Recoveries were 91.2% for norflurazon and 102% 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

<table>
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<th>RESULT</th>
<th>TIME (MIN)</th>
<th>TIME (SEC)</th>
<th>HEIGHT</th>
<th>SEP</th>
<th>WI/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DESMETHYL</td>
<td>0.1418</td>
<td>4.191</td>
<td>0.001072</td>
<td>1.7707</td>
<td>BV</td>
<td>6.10</td>
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<tr>
<td>3</td>
<td>NORF</td>
<td>0.3186</td>
<td>4.807</td>
<td>-0.003171</td>
<td>1.6607</td>
<td>VV</td>
<td>6.45</td>
</tr>
</tbody>
</table>

**TOTALS:** 0.4604 -0.0023 34314

**UNIDENT AREA:** 4458

**DETECTED PKS:** 4  
**REJECTED PKS:** 0

**DIVISOR:** 1.00000  
**MULTIPLIER:** 1.00000

**NOISE:** 125.0  
**OFFSET:** 63
APPENDIX IV.

REPRESENTATIVE STANDARD CURVES
AND CHROMATOGRAMS
Norflurazone

Figure 1. Chemical structures of norflurazone (4-chloro-5-(methylamino)-2-(α-α-α-trifluoro-m-tolyl)-3(2H)pyridazinone) and desmethyl norflurazone (5-amino-4-(chloro)-2-(α-α-α-trifluoro-m-tolyl)-3(2H)-pyridazinone).
Figure 2. Representative computer generated standard curves of norflurazon and desmethyl norflurazon.
Figure 3. Representative EC-GC chromatogram of a 0.005 ng/μL each of norflurazon and desmethyl norflurazon GC standard; 2 μL injected.
Figure 4. Representative GC/EC chromatogram of a 0.05 ng/dL each of norfuranaz and desmethyl norfuranaz standard; 2 ul injected.
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.
Figure 6. Representative GC chromatogram of peanut hulls check sample.

1.0 mg equivalent injected, < 0.01 ppm each of norflurazon and desmethyl norflurazon detected.

**Title:** NORF + DES IN PEANUTS  
**Date:** 19 APR 88  
**Time:** 9:20

**Sample:** CK HULLS #93  
**Method:** N+D1

<table>
<thead>
<tr>
<th>PEAK NO</th>
<th>PEAK NAME</th>
<th>RESULT</th>
<th>TIME (MIN)</th>
<th>OFFSET</th>
<th>HEIGHT</th>
<th>SEP</th>
<th>WI/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>DESMETHYL</td>
<td>0.0118</td>
<td>4.206</td>
<td>-0.014</td>
<td>1473</td>
<td>VB</td>
<td>3.45</td>
</tr>
<tr>
<td>4</td>
<td>NORFLURAZ</td>
<td>0.0162</td>
<td>4.842</td>
<td>0.002</td>
<td>1560</td>
<td>VV</td>
<td>?</td>
</tr>
</tbody>
</table>

**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 peanut green hay check sample and 0.01 ppm of desmethyl norflurazon detected.

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

Channel No: 1  Sample: CK GRHAY 84  Method: N+D1

Peak  Peak  Result  Time  Time  Height  Sep  W1/2
No  Name  (Min)  Offset  Counts  Code  (Sec)
1  DESMETHYL  0.0111  4.131  -0.099  1425  BB  3.70
4  NORFLURAZ  0.0468  4.059  -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 peanut dry hay check sample and <0.01 ppm of desmethyl norflurazon detected.

<table>
<thead>
<tr>
<th>PEAK</th>
<th>PEAK NAME</th>
<th>RESULT</th>
<th>TIME (MIN)</th>
<th>OFFSET</th>
<th>HEIGHT</th>
<th>SEP</th>
<th>WI/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DESMETHYL</td>
<td>0.0116</td>
<td>4.131</td>
<td>-0.089</td>
<td>1446</td>
<td>BB</td>
<td>3.90</td>
</tr>
<tr>
<td>3</td>
<td>NORFLURAZ</td>
<td>0.0692</td>
<td>4.844</td>
<td>0.004</td>
<td>6674</td>
<td>VV</td>
<td>7.15</td>
</tr>
</tbody>
</table>

TOTALS: 0.0808 -0.085 8120

IDENTIFIED AREA: 10143
DETECTED PKS: 0 REJECTED PKS: 1
DIVISOR: 1.00000 MULTIPLIER: 1.00000
NOISE: 85.0 OFFSET: 65
Representative EC-CC chromatogram of peanut meat check sample (96-0373).

Fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon; recoveries were 97.0% norflurazon and 102%
desmethyl norflurazon.

Figure 9.

<table>
<thead>
<tr>
<th>CHANNEL NO: 1</th>
<th>SAMPLE: CK+MEAT#93 3</th>
<th>METHOD: N+DI</th>
</tr>
</thead>
<tbody>
<tr>
<td>PEAK NO NAME</td>
<td>RESULT</td>
<td>TIME</td>
</tr>
<tr>
<td>1 DESMETHYL</td>
<td>0.2973</td>
<td>4.21</td>
</tr>
<tr>
<td>4 NORFLURAZ</td>
<td>0.3259</td>
<td>4.836</td>
</tr>
<tr>
<td>TOTALS:</td>
<td>0.6232</td>
<td>-0.013</td>
</tr>
</tbody>
</table>

UNIDENT AREA: 2519

DETECTED PKS: 7 REJECTED PKS: 0

DIVISOR: 1.00000 MULTIPLIER: 1.00000

NOISE: 85.0 OFFSET: -68
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.
Figure 11. Representative EC-GC chromatogram of peanut green hay check sample fortified at 0.1 ppm each of norflurazon and desmethyl norflurazon. Recoveries were 95.0% norflurazon and 83.0% desmethyl norflurazon.

<table>
<thead>
<tr>
<th>PEAK NO.</th>
<th>PEAK NAME</th>
<th>PEAK TIME</th>
<th>TIME OFFSET</th>
<th>HEIGHT SEPARATION</th>
<th>SEP CODE</th>
<th>W1/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DESMETHYL</td>
<td>0.2020</td>
<td>4.208</td>
<td>-0.022</td>
<td>26038</td>
<td>BV</td>
</tr>
<tr>
<td>4</td>
<td>NORFLURAZ</td>
<td>0.3011</td>
<td>4.838</td>
<td>-0.022</td>
<td>29046</td>
<td>VV</td>
</tr>
</tbody>
</table>

TOTALS: 0.5031 -0.044 55084

IDENT AREA: 6754
DETECTED PKS: 6 REJECTED PKS: 0
DIVISOR: 1.00000 MULTIPLIER: 1.00000
NOISE: 27.9 OFFSET: 26

REPORT NO. 1, PROJECT 40486

14:16 21 APR 88
Figure 12.
Representative GC chromatogram of peanut dry hay check sample fortified at 0.1 ppm each of norflurazon, 1.0 mg equivalent, was injected; recoveries were 99.0% norflurazon and 90.0% desmethyl norflurazon.

Title: NORF+DES IN PEANUTS
13:26 19 APR 88

Channel No: 1
Sample: CK+DRHY#01-1
Method: N+D1

Peak Peak Result Time Time Height Sep W1/2
No Name (MIN) Offset Counts Code (SEC)
1 DESMETHYL 0.2721 4.211 -0.009 326 33978 BV 4.95
3 NORFLURAZ 0.3840 4.835 -0.005 37632 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 13. Representative GC-EC chromatogram of peanut meat treatment sample (86-06-96) showing norflurazon and desmethylnorflurazon detected. 1.0 mg equivalent was injected. <0.01 ppm norflurazon and 0.032 ppm desmethylnorflurazon detected.

Title: NORF+DES IN PEANUTS

Channel No: 1  Sample: TR MEAT 96  Method: N+D1

Peak Name  Result Time  Time Offset  Counts  Code  Wt/2
1 Desmethyl  0.0925  4.201  -0.019  11545  BV  4.90
2 Norflurazon  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 14.
Representative EC-GC chromatogram of peanut hulls treatment sample and 0.74 desmethyl norflurazon detected.

TITLE: NORF+DES IN PEANUTS
15:14 21 APR 88

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

PEAK PEAK RESULT TIME TIME HEIGHT SEP W1/2
NO NAME (MIN) OFFSET COUNTS CODE (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

IDENTIFIED AREA: 3025

DETECTED PKS: 4  REJECTED PKS: 0

DIVISOR: 1.00000  MULTIPLIER: 1.00000

NOISE: 27.9  OFFSET: -12
### Figure 15

Representative EC-GC chromatogram of peanut green hay treatment sample (86-06786) showing peaks at 4.208, 4.834, 5.147, 5.438, and 5.762 minutes.

---

**Title:** NORF + DES in Peanuts

**Channel No.:** 1  
**Sample:** TR GRHAY B6  
**Method:** N+D1

<table>
<thead>
<tr>
<th>Peak Name</th>
<th>Result</th>
<th>Time (Min)</th>
<th>Time Offset</th>
<th>Height</th>
<th>Sep Code (SEC)</th>
<th>W1/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Desmethyl</td>
<td>0.1347</td>
<td>4.208</td>
<td>-0.012</td>
<td>16826</td>
<td>BV</td>
<td>4.90</td>
</tr>
<tr>
<td>Norfluraz</td>
<td>0.0823</td>
<td>4.834</td>
<td>-0.006</td>
<td>2934</td>
<td>VV</td>
<td>6.70</td>
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**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 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.

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>Log No.</th>
<th>Replication</th>
<th>Norflurazon</th>
<th>Desmethyl Norflurazon</th>
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<tbody>
<tr>
<td>Nut meat</td>
<td>86-06793</td>
<td>1</td>
<td>106</td>
<td>93</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>95</td>
<td>84</td>
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<tr>
<td></td>
<td></td>
<td>3</td>
<td>97</td>
<td>102</td>
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<tr>
<td></td>
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<td></td>
<td>$\bar{x} \pm sd$ 99.3 $\pm$ 5.9 93.0 $\pm$ 9.0</td>
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<tr>
<td>Hulls</td>
<td>86-06793</td>
<td>1</td>
<td>98</td>
<td>100</td>
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<tr>
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<td>2</td>
<td>101</td>
<td>104</td>
</tr>
<tr>
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<td>3</td>
<td>104</td>
<td>108</td>
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<tr>
<td></td>
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<td></td>
<td>$\bar{x} \pm sd$ 101.0 $\pm$ 3.0 104 $\pm$ 4.0</td>
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<tr>
<td>Green hay</td>
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<td>98</td>
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<tr>
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<td>83</td>
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<td>$\bar{x} \pm sd$ 100.3 $\pm$ 11.9 87.0 $\pm$ 9.6</td>
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<tr>
<td>Dry hay</td>
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<td>90</td>
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<td>3</td>
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<td>87</td>
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<tr>
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<td></td>
<td>$\bar{x} \pm sd$ 92.3 $\pm$ 9.9 87.5 $\pm$ 2.5</td>
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</tbody>
</table>