MEMORANDUM

SUBJECT: Multi-Residue Method Testing of Oxyfluorfen

FROM: Felecia A. Fort, Chemist
Reregistration Section II
Chemistry Branch II: Reregistration Support
Health Effects Division (H7509C)

THRU: William J. Hazel, Ph.D., Section Head
Reregistration Section II
Chemistry Branch II: Reregistration Support
Health Effects Division (H7509C)

TO: Harvey Hundley, Head
Analytical Chemistry Section
Analytical Chemistry Branch
Biological and Economic Analysis Division (H7503C)

Attached are the completed forms from the multi-residue method testing of Oxyfluorfen.

Chemical Name: 2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl)benzene

MRID No.: 422064-01

Multi-Residue Protocols Tested: A, D, E, and deleted Protocol II

Sponsor: Rohm and Haas Company

Performing Laboratory: QC Inc.
Southampton, PA

40 CFR Ref.: 180.381

If you have any questions please don’t hesitate to call.

Attachment: Multi-Residue Testing Forms
Attachment (1): Cited study (12 pages), MRID# 422064-01
cc (without attachment): Reviewer(F. Fort), C. Furlow(PIB/FOD), RF, SF, Circ.
RDI: WJHazel:3/30/92:EZager:4/10/92
H7509C:CBRS:CM#2:Rm800-E:305-7990:FAFort/FF:3/25/92
PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Appendix II
Reporting Form I

BEHAVIOR OF * Goal THROUGH PAM I METHOD 211.1/212.1/252

The following data resulted from testing the compound * Goal through PAM I methods 211.1/212.1/252, according to Appendix II, Protocol I.

Compound Name: * Goal
Alternate Names: 2-chloro-1-(1-ethoxy-4-nitrophenyl)-4-(trifluoromethyl) benzene

EPA Std. No.: \( C_{15} H_{11} NO_4 Cl F_3 \)
Molecular Formula:
Structure:

Comments: Common Name: oxyfluorfen

Results of Gas Chromatographic Tests

Standard reference material dissolved in iso-octane/hexane

OV-101 column (at conditions described in PAM I Table 331-A):

\[ R_t(c) \text{ for } p,p'\text{-DDT} : 3.20 \]
\[ R_t(c) \text{ for ethion} : 2.57 \]
\[ R_t(c) \text{ for } * \text{ Goal} : 2.00 \]

*OV-225 column (at conditions described in PAM I Table 331-F):

\[ R_t(c) \text{ for } p,p'\text{-DDT} : 3.79 \]
\[ R_t(c) \text{ for ethion} : 4.00 \]
\[ R_t(c) \text{ for } * \text{ Goal} : 4.08 \]

DEGS Column (at conditions described in PAM I Table 334-A):

\[ R_t(c) \text{ for parathion} : 2.62 \text{ ECD}, 2.25 \text{ TSD} \]
\[ R_t(c) \text{ for monocrotophos} : 4.62 \text{ ECD}, 4.40 \text{ TSD} \]
\[ R_t(c) \text{ for } * \text{ Goal} : 5.03 \text{ ECD}, 4.50 \text{ TSD} \]
OV-17 column (at conditions described in PAM I Table 331-G):
  \( k_{rt}(c) \) for \( p,p'-\text{DDT} \): 3.49
  \( k_{rt}(c) \) for \( \text{parathion} \): 1.09
  \( k_{rt}(c) \) for *Goal*: 2.13

Other column tested:

Conditions:
  \( k_{rt}(c) \) for \( p,p'-\text{DDT} \): ___
  \( k_{rt}(c) \) for \( \text{parathion} \): ___
  \( k_{rt}(c) \) for *: ___

\( ^{63} \text{Ni} \) electron capture detector (at conditions described in PAM I 311.4):

GLC column used: 5% OV-101
ng chlordane for 50% FSD: 1.0
ng *: Goal
  for 50% FSD: 1.3

Hall 700A (halogen) detector (at conditions described in PAM I 315):

GLC column used:
ng chlordane for 50% FSD:
ng *: Goal
  for 50% FSD:

FPD(P) detector (at conditions described in PAM I 314 and Table 311-G):

GLC column used:
ng chlordane for 50% FSD:
ng *: Goal
  for 50% FSD:

Other detector: Thermionic Specific (Nitrogen/Phosphorus)
  Conditions:

GLC column used: 5% OV-101
ng chlordane for 50% FSD: 1.5
ng *: Goal
  for 50% FSD: 30.

Recovery Through Florisil

Micrograms * Goal added to column: 100
Percent eluted with 6, 15, and 50% ethyl ether/pet ether (PAM I 211.14d):

<table>
<thead>
<tr>
<th>6%</th>
<th>15%</th>
<th>50%</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.10</td>
<td>0.22</td>
<td>&lt;0.10</td>
</tr>
<tr>
<td>108.</td>
<td>102.</td>
<td></td>
</tr>
</tbody>
</table>

TRANSMITTING NO. 87-1 (12/86)

FDBM FDA 2305A (7/84)
PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Percent eluted with Eluates 1, 2 and 3 (PAM I 252.12b):

1  0.12  0.20
2  0.02  0.14
3  0.50  0.60

For compounds which eluted in 65 eluate (211.14d) or Eluate 1 (252.12b) only:

Micrograms * Goal added to column: 300
Percent eluted with 250 ml petroleum ether forerun (per PAM I 211.14d and Table 201-X):

PE
6% ________ *not applicable*

PE
1 ________

Recovery Through Complete Method

Method for Fatty Foods

Fatty food sample: Milk
Analyzed for interferences? Yes
Reagent Blank analyzed for interferences? Yes

Method 211.13(n), 211.14a, d

Duplicate 100 g sample fortified at 0.05 ppm and at 0.5 ppm.
Percent recovered using 6, 15, and 50% ethyl ether/pet ether elution system:

<table>
<thead>
<tr>
<th></th>
<th>0.05 ppm</th>
<th>0.50 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Trial 1</td>
<td>Trial 2</td>
</tr>
<tr>
<td>6%</td>
<td>&lt;2.0</td>
<td>&lt;2.0</td>
</tr>
<tr>
<td>15%</td>
<td>&lt;2.0</td>
<td>&lt;2.0</td>
</tr>
<tr>
<td>50%</td>
<td>39.</td>
<td>28.</td>
</tr>
</tbody>
</table>

TRANSMITTAL NO 87-1 (12/86)
FORM FDA 2905a (7/86)
PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Method 211.13(b), 211.14a, 252

Duplicate 100 g sample fortified at 0.05 and at 0.50 ppm.
Percent recovered using Eluates 1, 2, and 3 of the methylene chloride elution system:

<table>
<thead>
<tr>
<th></th>
<th>0.05 ppm</th>
<th></th>
<th>0.5 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Trial 1</td>
<td>Trial 2</td>
<td>Trial 1</td>
</tr>
<tr>
<td>1</td>
<td>&lt;2.0</td>
<td>&lt;2.0</td>
<td>&lt;0.2</td>
</tr>
<tr>
<td>2</td>
<td>37.</td>
<td>21.</td>
<td>58.</td>
</tr>
<tr>
<td>3</td>
<td>&lt;2.0</td>
<td>&lt;2.0</td>
<td>&lt;0.2</td>
</tr>
</tbody>
</table>

Method for Nonfatty Foods
Nonfatty food sample: Apples
Analyzed for interferences? Yes
Reagent blank analyzed for interferences? Yes

Method 212.13(c), 211.14d

Duplicate 100 g analyzed at 0.05 ppm and at 0.50 ppm.
Percent recovered using ethyl ether/pet ether elution system:

<table>
<thead>
<tr>
<th></th>
<th>0.05 ppm</th>
<th></th>
<th>0.5 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Trial 1</td>
<td>Trial 2</td>
<td>Trial 1</td>
</tr>
<tr>
<td>6%</td>
<td>&lt;2.0</td>
<td>&lt;2.0</td>
<td>&lt;0.2</td>
</tr>
<tr>
<td>15%</td>
<td>&lt;2.0</td>
<td>&lt;2.0</td>
<td>&lt;0.2</td>
</tr>
<tr>
<td>50%</td>
<td>66.</td>
<td>79.</td>
<td>77.</td>
</tr>
</tbody>
</table>

Method 212.13(c), 252

Duplicate 100 g sample fortified at 0.05 ppm and 0.5 ppm.
Percent recovered using methylene chloride elution system:

<table>
<thead>
<tr>
<th></th>
<th>0.05 ppm</th>
<th></th>
<th>0.5 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Trial 1</td>
<td>Trial 2</td>
<td>Trial 1</td>
</tr>
<tr>
<td>1</td>
<td>&lt;2.0</td>
<td>&lt;2.0</td>
<td>&lt;0.2</td>
</tr>
<tr>
<td>2</td>
<td>87.</td>
<td>75.</td>
<td>86.</td>
</tr>
<tr>
<td>3</td>
<td>&lt;2.0</td>
<td>&lt;2.0</td>
<td>&lt;0.2</td>
</tr>
</tbody>
</table>

Information submitted by: QC Inc.
Address: 1205 Industrial Hwy., Southampton, Pa. 18966
Phone: (215) 355-3900
Date: 10/16/89

TRANSPORT/CAR NO 87-1 (12/86)
PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Other column tested: ____________________

Conditions:
Art(c) for ethion: ______

FPD (P) detector (at conditions described in PAM I 314 and Table 311-6):

GLC column used:
ng chlorpyrifos for 50% FSD:
ng * for 50% FSD: ______

Other detector: Thermionic Specific (Nitrogen/Phosphorus)

Conditions:
GLC column used: 5% OV-101
ng chlorpyrifos for 50% FSD: 1.5
ng * Goal For 50% FSD: 30

Recovery Through Charcoal Column:

Micrograms * Goal added to column: 50
Percent recovered from column per PAM I 232.34:

Trial 1: 54
Trial 2: 53

Recovery of Through Complete Method

Nonfatty food sample: Apples
Analyzed for interferences? Yes
Reagent blank analyzed for interferences? Yes

Duplicate 100 g sample fortified at 0.10 ppm and 0.50 ppm.
Percent recovered:

<table>
<thead>
<tr>
<th>0.10 ppm</th>
<th>0.50 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial 1</td>
<td>Trial 2</td>
</tr>
<tr>
<td>54</td>
<td>58</td>
</tr>
</tbody>
</table>

Information Submitted by: QC Inc.
Address: 1205 Industrial Hwy., Southampton, Pa. 18966
Phone: 215-355-3900
Date: 10/16/89

TR34-91-81

Appendix II
Reporting Form II

000025

FORM 4695 REV. 10/78
PESTICIDE ANALYTICAL MANUAL - VOLUME I  
Foods And Feeds

BEHAVIOR OF * Goal THROUGH PAM I METHOD 232.3

The following data resulted from testing the compound * Goal through PAM I method 232.3 (Storherr procedure), according to Appendix II Protocol II.

Compound Name: * Goal
Alternate Name: 2-chloro-1-(3-ethoxy-4-nitrophenoxyl)-4-(trifluoromethyl) benzene

EPA Std. No.: 

Molecular Formula: \( C_{15} H_{11} NO_4 Cl F_3 \)

Structure:

![Chemical Structure](image)

Comments: Common Name: oxyfluorfen

Results of Gas Chromatographic Tests

Standard reference material dissolved in iso octane/hexane

DEGS column (at conditions described in PAM I Table 334-A):

Rt(c) for parathion: 2.25 TSD
Rt(c) for monocrotophos: 4.40 TSD
Rt(c) for * Goal: 14.50 TSD

* OV-225 column (at conditions described in PAM I Table 331-F):

Rt(c) for ethion: column not compatible with nitrogen selective detectors.

OV-17 column (at conditions described in PAM I Table 331-G):

Rt(c) for ethion: 3.00 TSD
Rt(c) for * Goal: 1.75 TSD

OV-101 column (at conditions described in PAM I Table 331-A):

Rt(c) for ethion: 2.22
Rt(c) for * Goal: 1.67
PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Appendix II
Reporting Form III

BEHAVIOR OF Goal THROUGH PAM I 232.4

The following data resulted from testing the compound Goal through
PAM I method 232.4 (Luke procedure), according to PAM I Appendix II, Protocol
III.

Compound Name: Goal

Alternate Names: 2-chloro-1-(3-ethoxy-4-nitrophenox)-4-(trifluoromethyl) benzene

EPA Std. No.: 

Molecular Formula: C₁₅ H₁₁ NO₄ Cl F₃

Structure:

![Chemical Structure]

Comments: Common Name: oxyfluorfen

Results of Gas Chromatographic Tests

Standard reference material dissolved in iso octane/hexane

OV-101 column (at conditions described in PAM I Table 331-A):

Rt(c) for p,p'-DDT: 3.20
Rt(c) for ethion: 2.57
Rt(c) for Goal: 2.00

OV-17 column (at conditions described in PAM I Table 331-G):

Rt(c) for p,p'-DDT: 3.49
Rt(c) for ethion: 3.40
Rt(c) for Goal: 2.13

*OV-225 column (at conditions described in PAM I Table 331-F):

Rt(c) for p,p'-DDT: 3.79
Rt(c) for ethion: 4.00
Rt(c) for Goal: 4.08

*This column not compatible with nitrogen selective detectors.

TRANSMITTAL NO 87-1 (12/85)

FORM FDA 2905a (7/84)
DEGS column (at conditions described in PAM I Table 334-A):

Rt(c) for parathion: 2.62 ECD
Rt(c) for monocrotophos: 4.62 ECD
Rt(c) for * Goal: 5.01 ECD

Ultrabond 20 SE:

Rt(c) for parathion: 
Rt(c) for monocrotophos: 
Rt(c) for *

Other column:

Conditions:

Rt(c) for parathion: 
Rt(c) for *

Hewlett Packard 700 A (halogen) detector (at conditions described in PAM I 315):

GLC column used:
ng chlorpyrifos for 50% FSD: 
ng * for 50% FSD:

FPD (P) detector (at conditions described in PAM I Tables 334-A, 331-G):

GLC column used:
ng chlorpyrifos for 50% FSD: 
ng * for 50% FSD:

63Ni electron capture detector (at conditions described in PAM I 311.4):

GLC column used: 5% OV-101
ng chlorpyrifos for 50% FSD: 1.5
ng * Goal: 50% FSD: 30.

Other detector(s) tested: Thermionic Specific (Nitrogen/Phosphorus)
Conditions:

GLC column used: 5% OV-101
ng chlorpyrifos for 50% FSD: 1.5
ng * Goal: 50% FSD: 30.
Recovery Through Method

Non-fatty food sample: Apples
Analyzed for interferences? Yes
Reagent blank analyzed for interferences? Yes

Duplicate 100 g sample fortified at 0.1 ppm and 0.5 ppm.
Percent recovered:

<table>
<thead>
<tr>
<th></th>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 1</th>
<th>Trial 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1 ppm</td>
<td>110</td>
<td>88</td>
<td>96</td>
<td>98</td>
</tr>
<tr>
<td>0.5 ppm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

[Report results of studies using Florisil cleanup with 15% ethyl ether/petroleum ether cleanup, as in PAM I 212.2, if the compound is determined by electron capture detector.]

Information Submitted By: QC Inc.
Address: 1205 Industrial Dr., Southampton, Pa. 18966
Phone: (215) 355-3900
Date: 10/16/89
PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Appendix II
Reporting Form IV

BEHAVIOR OF * Goal THROUGH PAM I 242.1
and JAOAC (1985) 68. 726-733

The following data resulted from testing the compound * Goal through
the method described in PAM I 242.1 and JAOAC (1985) 68 726-733, according to
PAM I Appendix II, Protocol IV. (Also see AOAC Methods of Analysis Sec.

Compound Name: * Goal

Alternate Names: 2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl) benzene

EPA Std. No.:

Molecular Formula: $C_{15}H_{11}NO_4ClF_3$

Structure:

Chemical Properties:

Usage:

Comments: Common Name: * nxyfluorfen

Results of High Performance Liquid Chromatographic Tests

HPLC post-column (fluorometric labeling determinative system described in method
used with the following columns:

Analytical Column:

Guard Column: *Not applicable
PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Natural Fluorescence: Detector described in J. Chromatog. (1983) 225 497-510;
fluoresces at excitation and emission wavelengths of ______ and ______ nm, respectively.

<table>
<thead>
<tr>
<th>Natural Fluorescence</th>
<th>Post-Column Fluorometric Labeling</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peak Shape</td>
<td>*Not applicable</td>
</tr>
<tr>
<td>Retention time (rel. carbofuran)</td>
<td></td>
</tr>
<tr>
<td>ng causing 50% FSD</td>
<td></td>
</tr>
<tr>
<td>Linear Range</td>
<td></td>
</tr>
</tbody>
</table>

[NOTE: If GLC characteristics of compound were determined, report these on copies of appropriate Reporting Forms I-III.]

**Results of Stability in Methanol Studies**

<table>
<thead>
<tr>
<th>Short Term Study</th>
<th>Long Term Study</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>Day</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

**Recovery Through Charcoal-Silanized Celite Column**

Micrograms * Goal added to column: 25.
Percent recovered from charcoal-silanized Celite column:

<table>
<thead>
<tr>
<th>Methylene chloride + toluene-acetonitrile</th>
<th>Additional 100 ml toluene-acetonitrile</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial 1</td>
<td>Trial 2</td>
</tr>
<tr>
<td>73.</td>
<td>&lt;1.0</td>
</tr>
<tr>
<td>79.</td>
<td>1.8</td>
</tr>
</tbody>
</table>

TRANSMITTAL NO  87-1 (12/86)
FORM FDA 2905a (7/86)
PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Recovery Through Complete Method

Food sample: Apples
Analyzed for interferences? Yes
Reagent blank analyzed for interferences? Yes

Determinative step used for recovery test: GLC

Duplicate 150 g samples fortified at 0.05 ppm and 0.25 ppm.

Percent recovered:

<table>
<thead>
<tr>
<th></th>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 1</th>
<th>Trial 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05 ppm</td>
<td>101.</td>
<td>88.</td>
<td>110.</td>
<td>104.</td>
</tr>
</tbody>
</table>

Additional Data on crop used as samples:

Pesticide residues found:

Unidentified peaks (specify determinative step used and list peaks by retention time relative to appropriate compound).

Information submitted by: QC Inc.
Address: 1205 Industrial Hwy., Southampton, Pa. 18966
Phone: (215) 355-3900
Date: 10/16/89