



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

APR 16 1992

OFFICE OF
PESTICIDES AND TOXIC
SUBSTANCES

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)

Felecia Fort

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

W. J. Hazel

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



Printed on Recycled Paper

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 FeedsAppendix II
Reporting Form IBEHAVIOR OF * Goal
PAM I METHOD 211.1/212.1/252

THROUGH

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

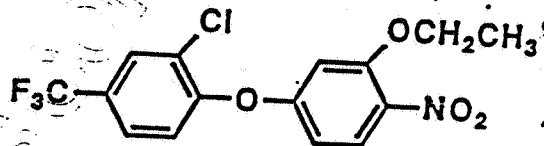
Compound Name: * Goal

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

EPA Std. No.: C₁₅ H₁₁ NO₄ Cl F₃

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):

Rrt(c) for p,p'-DDT: 3.20

Rrt(c) for ethion: 2.57

Rrt(c) for * Goal : 2.00

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

Rrt(c) for p,p'-DDT: 3.79

Rrt(c) for ethion: 4.00

Rrt(c) for * Goal : 4.08

*This column not compatible with
nitrogen selective detectors.

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

Rrt(c) for parathion: 2.62 ECD, 2.25 TSD

Rrt(c) for monocrotophos: 4.62 ECD, 4.40 TSD

Rrt(c) for * Goal : 5.03 ECD, 4.50 TSD

TRANSMITTAL NO

87-1 (12/86)

FORM FDA 2905a (7/86)

TR34-91-81

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PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Appendix II
Reporting Form I

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

Rrt(c) for p,p'-DDT: 3.49
Rrt(c) for parathion: 1.09
Rrt(c) for *Goal : 2.13

Other column tested: _____
Conditions: _____

Rrt(c) for p,p'-DDT: _____
Rrt(c) for parathion: _____
Rrt(c) for * : _____

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 For 50% FSD: 1.2

Hall 700A (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 314 and Table 311-G):

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 Florisil

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

6%	<u><0.10</u>	<0.10
15%	<u>0.22</u>	0.22
50%	<u>108.</u>	102.

TRANSMITTAL NO

87-1 (12/86)

15

FORM FDA 2905a (7/86)

4

000023

TR34-91-81

PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Appendix II
Reporting Form I

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

1	0.12	0.20
2	102.	117.
3	0.50	0.60

For compounds which eluted in 6% eluate (211.14d) or Eluate 1 (252.12b) only:

Micrograms * Goal added to column: 100
Percent eluted with 250 ml petroleum ether forerun (per PAM I 211.14d and Table 201-K):

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(h), 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/petr ether elution system:

	0.05 ppm		0.50 ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
6%	<2.0	<2.0	<0.2	<0.2
15%	<2.0	<2.0	<0.2	<0.2
50%	39.	28.	58.	65.4

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87-1 (12/86)

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PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And FeedsAppendix II
Reporting Form IMethod 211.13(h), 211.14a, 252

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

	0.05 ppm		0.5 ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
1	<2.0	<2.0	<0.2	<0.2
2	37.	21.	58.	58.
3	<2.0	<2.0	<0.2	<0.2

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/petr ether elution system:

	0.05 ppm		0.50 ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
6%	<2.0	<2.0	<0.2	<0.2
15%	<2.0	<2.0	<0.2	<0.2
50%	66.	79.	77.	83.

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:

	0.05 ppm		0.5 ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
1	<2.0	<2.0	<0.2	<0.2
2	67.	75.	66.	74.
3	<2.0	<2.0	<0.2	<0.2

Information submitted by:

Address:

QC Inc.

1205 Industrial Hwy., Southampton, Pa. 18966

Phone: (215) 355-3900

Date: 10/16/89

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87-1 (12/86)

FORM PIA 2905a (7/86)

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TR34-91-81

PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And FeedsAppendix II
Reporting Form IIOther column tested: _____
Conditions: _____Rrt(c) for ethion: _____
Rrt(c) for * : _____

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

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 ColumnMicrograms * Goal added to column: 50.
Percent recovered from column per PAM I 232.34:Trial 1: 54.
Trial 2: 53.Recovery of Through Complete MethodNonfatty food sample: Apples
Analyzed for interferences? Yes
Reagent blank analyzed for interferences? YesDuplicate 100 g sample fortified at 0.10 ppm and 0.50 ppm.
Percent recovered:

0.10 ppm		0.50 ppm	
Trial 1	Trial 2	Trial 1	Trial 2
44.	58.	64.	38.

Information submitted by: QC Inc.
Address: 1205 Industrial Hwy., Southampton, Pa. 18966Phone: 610 355-3900
Date: 10/18/89

TRANSMITTED NO 87-1 (12/87)

FORM PDA 2905a (7/86)

24

7

TR34-91-81

000026

PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Appendix II
Reporting Form II

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.

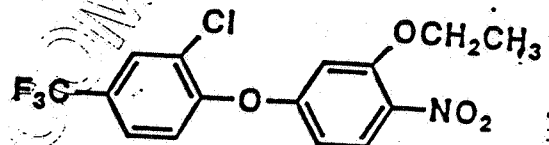
Compound Name: * Goal

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

EPA Std. No.:

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

Structure:



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):

Rrt(c) for parathion: 2.25 TSD
Rrt(c) for monocrotophos: 4.40 TSD
Rrt(c) for * Goal: 4.50 TSD

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

Rrt(c) for ethion: _____
Rrt(c) for _____ : _____ *column not compatible with nitrogen selective detectors.

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

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

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

Rrt(c) for ethion: 2.22
Rrt(c) for * Goal: 1.67

TRANSMITTAL NO 87-1 (12/87)

FORM FDA 2905a (7/86)

PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And FeedsAppendix I
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.

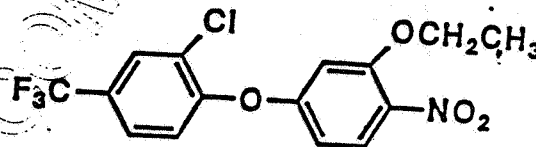
Compound Name: * Goal

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

EPA Std. No.:

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

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):

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

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

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

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

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

*This column not compatible with
nitrogen selective detectors.

TRANSMITTAL NO

87-1 (12/86)

FORM FDA 2905a (7/86)

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TR34-91-81

PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And FeedsAppendix II
Reporting Form III

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

Rrt(c) for parathion: 2.62 ECD
 Rrt(c) for monocrotophos: 4.62 ECD
 Rrt(c) for * Goal : 5.03 ECD

Ultrabond 20 SE:

Rrt(c) for parathion: _____
 Rrt(c) for monocrotophos: _____
 Rrt(c) for * : _____

Other column: _____
 Conditions: _____

Rrt(c) for parathion: _____
 Rrt(c) for * : _____

Hall 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 for 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 for 50% FSD: 30.

TRANSMITTAL NO

87-1 (12/85)

30

FORM 70A 2905a (7/86)

10

000029

TR34-91-81

PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And FeedsAppendix 11
Reporting Form 111Recovery Through MethodNonfatty food sample: Apples
Analyzed for interferences? Yes
Reagent blank analyzed for interferences? YesDuplicate 100 g sample fortified at 0.1 ppm and 0.5 ppm.
Percent * recovered:

	0.1 ppm	0.5 ppm	
Trial 1	Trial 2	Trial 1	Trial 2
110.	88.	98.	98.

[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 Hwy., Southampton, Pa. 18966Phone: (215) 355-3900
Date: 10/16/89

TRANSMITTAL NO

87-1 (12/86)

FORM FDA 2503a (7/86)

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TR34-91-81

PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And FeedsAppendix II
Reporting Form IVBEHAVIOR 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. 29.A05-29.A13.)

Compound Name: * Goal

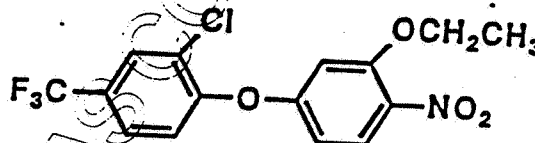
Alternate Names: 2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl) benzene

EPA Std. No.:

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

Structure:

Chemical Properties:



Usage:

Comments: Common Name: oxyfluorfen

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

TRANSMITTAL NO

87-1 (12/86)

FORM FDA 2905a (7/86)

12

TR34-91-81

000031

PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Appendix II
Reporting Form IV

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

	Natural Fluorescence	Post-Column Fluorometric Labeling	
Peak Shape	_____	_____	*Not applicable
Retention time (rel. carbofuran)	_____	_____	
ng causing 50% FSD	_____	_____	
Linear Range	_____	_____	

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

Results of Stability in Methanol Studies

Short Term Study		Long Term Study		
Time	Peak Ht (mm)	Day	Peak Ht (mm)	
_____	_____	_____	_____	*Not applicable
_____	_____	_____	_____	
_____	_____	_____	_____	
_____	_____	_____	_____	
_____	_____	_____	_____	
_____	_____	_____	_____	

Recovery Through Charcoal-Silanized Celite Column

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

Methylene chloride +
toluene-acetonitrile

Additional 100 ml
toluene-acetonitrile

Trial 1	Trial 2
73.	79.

Trial 1	Trial 2
<1.0	1.8

TRANSMITTAL NO

87-1 (12/86)

FORM FDA 2905a (7/86)

TR34-91-81

000032

PESTICIDE ANALYTICAL MANUAL - VOLUME 1
Foods And Feeds

Appendix II
Reporting Form IV

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:

0.05 ppm		0.25 ppm	
Trial 1	Trial 2	Trial 1	Trial 2
101.	88.	110.	104.

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

39

TRANSMITTAL NO

87-1 (12/86)

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14