

Study Title

Addendum 2

A Gas Chromatographic Method for the Determination of
SENCOR and Metabolites in Various Crops

Method Update

Data Requirement

EPA Ref: 171-4(c), Residue Analytical Method - Crops

Author

J. S. Thornton

Original Study
Completion Date
June 14, 1974

Revised Date
April 15, 1994

Revisions
Prepared by
R. R. Gronberg

Performed by

Morse Laboratories Inc.
1525 Fulton Ave.
Sacramento, CA 95825

and

Chemonics Industries
734A Southern Pacific Drive
Phoenix, AZ 85034

Submitted by

Miles Inc.
Agricultural Division
P. O. Box 4913, Hawthorn Road
Kansas City, MO 64120

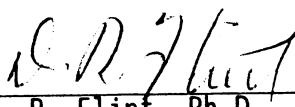
Miles Report Number

40901R-2

Data Confidentiality Statement

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA #10(d)(1)(A), (B) or (C).

Company: Miles Inc.
Agriculture Division
Research and Development Department
Environmental Research Section

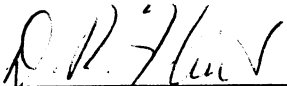
Company Agent:  Date: 4-19-94
D. R. Flint, Ph.D.
Director, Environmental Research

These data are the property of the Agricultural Chemicals Division of Miles Inc, and as such, are considered to be confidential for all purposes other than compliance with FIFRA 10. Submission of these data in compliance with FIFRA does not constitute a waiver of any right to confidentiality which may exist under any other statute or in any other country.

Certification of Good Laboratory Practice


The good laboratory practice requirements of 40 CFR Part 160 do not apply to the study described in this document.

Submitter: Miles Inc.
Agriculture Division
Research and Development Department
Environmental Research Section



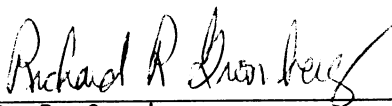
D. R. Flint, Ph.D.
Director, Environmental Research

Date: 4-19-94

Sponsor Representative: 

J. J. Murphy, Ph.D.
Manager, Residue Chemistry

Date: 4/18/94

Preparer: 

R. R. Gronberg
Assoc. Research Scientist

Date: 4/15/94

Certification of Authenticity

Preparer: Richard R Gronberg Date: 4/15/94
R. R. Gronberg
Assoc. Research Scientist

Approved By: J. J. Murphy Date: 4/18/94
J. J. Murphy, Ph.D.
Manager, Residue Chemistry

D. R. Flint Date: 4-19-94
D. R. Flint, Ph.D.
Director, Environmental Research

Inquiries

Inquiries should be directed to:

J. J. Murphy, Ph.D.
Miles Inc.
Agriculture Division
Miles Research Park
17745 South Metcalf Ave
Stilwell, KS 66085

Telephone: 913-897-9330

Table of Contents

	<u>Page No.</u>
Title Page.....	1
Data Confidentiality Statement.....	2
Certification of Good Laboratory Practice.....	3
Certification of Authenticity.....	4
Inquiries.....	4
Table of Contents.....	5
1.0 Introduction..	6
2.0 Analytical Method.....	6
2.1 Detailed Additions/Modifications.....	6
2.1.1 Gas Chromatographic Analysis.....	6
2.1.1.1 Alternate Columns, Conditions and Matrix.....	6
2.1.1.2 Alternate Detectors and Conditions.....	7
3.0 Conclusion.....	8
Appendices.....	9
1. Letter from R. J. Taylor to J. S. Thornton.....	9

Addendum 2

A Gas Chromatographic Method for the Determination of
Sencor and Metabolites in Various Crops

Method Update

1.0 Introduction

In the November, 1989, letter (See Appendix 1) from Mr. R. J. Taylor (EPA) to Mr. J. S. Thornton (Miles Inc), the Agency requested that all modifications to the original analytical method (Miles Ag Div Report No. 40901, MRID No. 00069067) should be submitted.

In a meeting which was held in January, 1994 with EPA CBRS personnel (Ms. Susan Hummel and Mr. Bart Suhre), Miles was again requested to submit an updated method which would include all the modifications to the enforcement method for the analysis of metribuzin and metabolites.

This report was prepared to provide a response to this EPA request.

2.0 Analytical Method

The analytical residue method for the analysis of SENCOR and metabolite residues is described in Miles Ag Div Report No. 40901, dated June, 1974 (later published in J. Agric. Food Chem., Vol 25, No. 2, 1977). This method, used for the analysis of alfalfa, asparagus, barley, corn, pea, wheat, processed products of tomato and sugarcane, and other various crops had few modifications through 1988.

These modifications were, primarily, the use of different gc column packing materials or the use of "nitrogen specific" detectors.

2.1 Detailed Additions/Modifications

2.1.1 GAS CHROMATOGRAPHIC ANALYSIS

2.1.1.1 Alternate Columns, Conditions and Matrix

1. Packing: 5% EGSS-X on 100/120 mesh GC-Q
- Size: 1/4 inches o.d. x 4 feet (glass)
- Temp.: Metribuzin: 180°C, 200°C, or 207°C
- DADK/DK: 195-200°C
- DA: 205-214°C
- Matrix: Alfalfa, Asparagus, Barley, Carrots,
Corn, Peas, Potatoes, Radishes, Sorghum,
Soybean, Sugarbeets, Tomatoes, Wheat.

2. Packing: 1% Hi EFF8BP on 100/120 mesh GC-Q
 Size: 1/4 inches o.d. x 4 feet (glass)
 Temp.: Metribuzin: 192°C
 DA: 205°C - 210°C
 Matrix: Alfalfa, Barley, Carrots, Corn, Peas,
 Potatoes, Radishes, Sorghum, Soybean,
 Sugarbeets, Tomatoes, Wheat.
3. Packing: 6% FFAP on 60/80 mesh GC-Q
 Size: 1/4 inches o.d. x 4 feet (glass)
 Temp.: DA: 220°C
 Matrix: Barley
4. Packing: 10% DC-200/1.5% QF-1 on 80/100 mesh GC-Q
 Size: 1/4 inches o.d. x 4 feet (glass)
 Temp.: Metribuzin: 172°C
 Matrix: Barley
5. Packing: 10% DC-200/1.5% QF-1 on 80/100 mesh Chromosorb W(HP)
 Size: 2 mm i.d. x 31 cm (glass)
 Temp.: Metribuzin: 175°C
 Matrix: Potatoes
6. Packing: 5% OV-225 on 80/100 mesh Chromosorb W(HP)
 Size: 2 mm i.d. x 34 cm (glass)
 Temp.: Metribuzin: 190°C
 DADK: 160°C for 1 min, 25°C/min
 to 185°C, hold for 8 min.
 or
 170°C for 2 min, 25°C/min
 to 185°C, hold for 2 min
 DK/DA: 160°C for 1 min, 25°C/min
 to 210°C, hold for 8 min.
 DA: 205°C, 220°C
 Matrix: Potatoes

2.1.1.2 Alternate Detectors and Conditions

1. Electrolytic Conductivity Detector (ELCD)
 Coulson N-cell detector or Tracor 560:700A or Equivalent.

Conditions:

Carrier Gas: 25 cc/min (helium)
 Detector Temp: 250°C
 Reaction Gas: 100 cc/min
 Conductivity solvent: 0.5 ml/min (N-proponal/DI water 1:1)
 Reactor: Nickel (with nitrogen mode scrubber)
 Reactor Temp: 900°C

2. Thermionic Specific Detector (TSD)
Tracor 560:702 N/P or Equivalent

Conditions:

Carrier Gas:	Helium or nitrogen
Carrier Flow:	Packed column: 30 cc/min
Detector Temp:	300°C
Air:	175 ml/min
Hydrogen:	4.5 ml/min (Optimum 4.0 to 5.5)

3.0 Conclusion

The above stated information, when taken along with the information in Miles Ag Div Report No. 40901, represents an updated method description for the determination of SENCOR residues in various crops.

Appendix 1. Letter from Mr. R. J. Taylor to Mr. J. S. Thornton



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

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

NOV 14 1989

Mr. John S. Thornton
Mobay Corporation
Agricultural Chemical Division
Hawthorn Road
P.O. Box 4913
Kansas City, MO 64120-0013

Dear Mr. Thornton:

Subject: Sencor 4 Flowable Herbicide
EPA Registration No. 3125-314
Sencor DF 75% Dry Flowable Herbicide
EPA Registration No. 3125-325
EPA Pesticide Petition No. 8F3683
EPA Food Additive Petition No. 8H5563
Your Application Dated August 11, 1988

This refers to Pesticide Petition No. 8F3683 which proposes the establishment of tolerances for the combined residues of the herbicide metribuzin (4-amino-6-(1,1-dimethylethyl)-3-(methylthio)-1,2,4-triazin-5-(4H)-one and its triazinone metabolites in or on the raw agricultural commodities alfalfa, seed at 0.1 part per million (ppm); alfalfa, chaff at 1.0 ppm; asparagus at 0.1 ppm; barley, forage at 2.0 ppm; barley, hay at 7.0 ppm; corn, silage at 0.1 ppm; corn, fresh, cannery waste at 0.1 ppm; pea straw at 4.0 ppm; wheat, hay at 7.0 ppm; and Food Additive Petition No. 8H5563 proposing tolerances for metribuzin and its triazinone metabolite in or on the processed commodities tomato, processed products at 0.2 ppm and sugarcane molasses at 2.0 ppm.

The scientific review and evaluation of the subject petitions have been completed. The following are our conclusions and/or comments.

Appendix 1. (Continued)

-3-

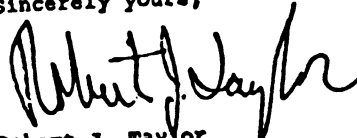
4. Methodology

- a. Analytical methods available for enforcement appear in PAM II. However, it is not clear that the extraction procedures of either the method described in Mobay Report No. 40901, for plant analysis, or Report No. 42257, for animal tissue analysis, will release triazinone metabolites to the same extent as the extraction procedures in the metabolism studies. The radiolabeled-treated wheat straw and soybean tissue should be analyzed using method 40901 and compare results to those from the respective metabolism studies.
- b. The residue analytical method for plants was only generally described in the petition. All modifications to the original method for each of the crops analyzed in this petition should be submitted.

-5-

Further action will await reply to the above comments concerning residue data and resolution of questions concerning developmental toxicity.

Sincerely yours,



Robert J. Taylor
Product Manager (25)
Fungicide-Herbicide Branch
Registration Division (M7505C)