UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

JUN 21 1991

CERTIFIED MAIL

Barbara Gingher, Ph.D.
Product Registration Manager
U.S. Regulatory Affairs
American Cyanamid Company
Agricultural Research Division
P.O. Box 400
Princeton, NJ 08540

Subject: Pendimethalin Registration Standard - Methodology and Product Chemistry Review

Dear Dr. Gingher:

The Agency has completed its review of data submitted for method validation for soybean forage (GLN 171-4C; MRID 41431001) and product chemistry (GLNs 62-2 and 62-3; MRID 41725201). A copy of the review is enclosed. Please note the following points from the review:

171-4C RESIDUE ANALYTICAL METHOD (REQUIRED TO VALIDATE RESIDUE DATA ON SOYBEAN FOLIAGE)

- The submitted method validation data for soybean forage (MRID 41431001) are not sufficient to support the available data (MRID 40185101) pertaining to residues of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite in or on soybean hay and straw. Additional data are required concerning method recoveries of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite from fortified samples of hay and straw analyzed by method M-1609.

  The additional data are due by 12/31/91.

- Currently, the qualitative nature of the residue in plants is not adequately understood. If the required metabolism studies reveal the presence of additional metabolites of concern, additional validated methods for data collection and tolerance enforcement will be required.

(1)

CONCURRENCES								
SYMBOL	H7508W	H7508W						
SURNAME	T. Stowe	Wedley						
DATE	06/11/91	6/20/91						

EPA Form 1320-1 (12-70) *U.S.GPO:1989-624-485/10186 OFFICIAL FILE COPY

62-2 CERTIFICATION OF LIMITS

- The data (MRID 41725201) regarding certified limits for American Cyanamid 90% T (EPA Reg. No. 241-245) do not satisfy the requirements of Guideline Reference No. 62-2 because a nominal concentration for the active ingredient was not included on the Confidential Statement of Formula (CSF).

  A complete CSF, submitted on EPA Form 8570-4 (Rev. 2-85), is required to fulfill the outstanding data requirements. **The additional data are due by 08/31/91.**

62-3 ENFORCEMENT ANALYTICAL METHODS

- The additional validation data (MRID 41725201) satisfy the data requirements for Guideline Reference No. 62-3. No additional data are required.

Within thirty (30) days of receipt of this letter, you must submit in writing a commitment to provide the additional information within the timeframes noted above. Failure to adequately respond within 30 days may result in the issuance of a Notice of Intent to Suspend affecting your Pendimethalin registrations.

If you have any further questions, please contact Terri Stowe of my staff at (703) 308 - 8043.

Sincerely yours,

/S/

Lois Rossi, Chief
Reregistration Branch
Special Review and
Reregistration Division

Enclosure

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

OFFICE OF
PESTICIDES AND TOXIC
SUBSTANCES

APR 24 1991

MEMORANDUM

SUBJECT: American Cyanamide Company: Response to the Pendimethalin Reregistration Standard: Methodology and Product Chemistry (MRID #'s 41431001 and 41725201, CBRS #'s 7507 and 7517, Barcodes: D159827 and D159905).

FROM: E. Zager, Chief
Chemistry Branch II: Reregistration Support
Health Effects Division (H7509C)

Debra Edwards, for

TO: Lois Rossi, Chief
Reregistration Branch
Special Review & Reregistration Division (H7508C)

and

Reto Engler, Ph.D., Chief
Science Analysis and Coordination Branch
Health Effects Division (H7509C)

Attached are two reviews of residue and product responses to the pendimethalin reregistration standard submitted by American Cyanamide Company. These reviews were completed by Dynamac Corporation under supervision of CBRS, HED. They have undergone secondary review in the branch and have been revised to reflect Agency policies.

Revised product chemistry data requirement tables are included.

If you need additional input please advise.

Attachment 1: Pendamethalin Residue Chemistry Review.

Attachment 2: Pendimethalin Product Chemistry Review.

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Attachment 3: Confidential Appendices A and B For American Cyanamide Company's Pendimethalin Product(s)

cc (With Attachments 1, 2 and 3): RBP, Pendimethalin Reregistration Standard file, Pendimethalin Subject File, C. Furlow/J. Burrell (PIB/FOD) and Dynamac.

cc (With Attachments 1 and 2) : Circulation (7).

cc (Without Attachments): RF.

ATTACHMENT 1

DYNAMAC
CORPORATION

Final Report

PENDIMETHALIN (CBRS No. 7507) Task 4: Registrant's Response to Residue Chemistry Data Requirements

April 8, 1991

Contract No. 68-D8-0080

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

Submitted by:
Dynamac Corporation
The Dynamac Building
2275 Research Boulevard
Rockville, MD 20850-3268

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PENDIMETHALIN (CBRS No. 7507)

REGISTRANT'S RESPONSE TO RESIDUE CHEMISTRY DATA REQUIREMENTS

Task - 4

BACKGROUND

The Pendimethalin Guidance Document (dated 3/29/85), requires additional data depicting residues of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite in or on soybean hay and straw. In response to soybean residue data requirements, American Cyanamid Company submitted data (MRID 40185101) pertaining to residues of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite in or on soybean hay and straw. These data were reviewed and conditionally accepted by CBRS (D. Edwards, EPA Memorandum CBRS No. 5495, dated 9/8/89) pending the receipt of method recovery data and a description of gas-chromatographic analytical method, Cyanamid Report M-1609. The Pendimethalin Residue Chemistry Registration Standard Update was issued on 3/19/90. We note that CBRS has concluded (CBRS Nos. 6570, 6603, 6604, and 7153, R. Loranger and R. Perfetti, dated 1/29/91) that the nature of the residue is not understood in plants.

In response to SRRD notification (3/6/90) of soybean data deficiencies in MRID 40185101, American Cyanamid has submitted (CBRS No. 7507) data (1990; MRID 41431001) containing a description of the gas-chromatographic method, American Cyanamid Report M-1609 and supporting validation data for soybean forage (Report C-2667).

The current submission is reviewed here for its adequacy in supporting the available soybean hay and straw residue studies.

DEFICIENCIES REMAINING TO BE RESOLVED

The remaining residue chemistry data gaps that are identified in the Pendimethalin Residue Chemistry Registration Standard Update (dated 3/19/90) and the Registrant's Response to the Residue Chemistry Data Requirements for Pendimethalin (CBRS No. 6570, 6603, 6604 and 7153; dated 1/29/91) have not been addressed in this submission and are still unresolved. The Conclusions and Recommendations stated below apply only to the soybean forage residue validation data contained in this submission. All other data gaps required in the Pendimethalin Residue Chemistry Registration Standard Update dated 3/19/90 remain outstanding.

CONCLUSIONS

1. Method recoveries of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite using American Cyanamid Method M-1609 (GC/FID) were 83.7%-104.8% and 83-112.7%, respectively, from 11-12 soybean forage samples fortified separately with each compound at 0.05-1 ppm. Recovery of the 3,5-dinitrobenzyl alcohol metabolite was 69.4% from one sample fortified at 0.1 ppm.

2. The submitted validation data for soybean forage do not adequately support the available residue data pertaining to the combined residues of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite in or on soybean hay and straw. Validation data requirements for these commodities remain outstanding.

RECOMMENDATIONS

The registrant should be given a copy of this report and informed of the following:

1. The submitted method validation data for soybean forage (MRID 41431001) are not sufficient to support the available data (MRID 40185101) pertaining to residues of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite in or on soybean hay and straw. Additional data are required concerning method recoveries of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite from fortified samples of hay and straw analyzed by method M-1609.

2. Currently, the qualitative nature of the residue in plants is not adequately understood (Registrant's Response to the Residue Chemistry Data Requirements for Pendimethalin; CBRS No. 6570, 6603, 6604 and 7153; dated 1/29/91). If the required metabolism studies reveal the presence of additional metabolites of concern, additional validated methods for data collection and tolerance enforcement will be required.

DETAILED CONSIDERATIONS

Residue Analytical Method

American Cyanamid Co. (1990; MRID 41431001) submitted a gas-liquid chromatographic method employing flame ionization detection (GLC/FID) for the determination of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite (4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol) in or on soybean forage (American Cyanamid Method M-1609). Samples are homogenized in methanol. The homogenate is filtered and the filtrate is concentrated by rotatory evaporation. The

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concentrate is partitioned between hexane and water and the aqueous phase is re-extracted with hexane. Hexane-soluble residues in the combined hexane fractions are evaporated to dryness, redissolved in hexane, and cleaned up on a Florisil solid phase extraction (SPE) column. Pendimethalin is eluted with sequential additions of hexane and 50:50 (v/v) hexane:dichloromethane; the combined eluates are evaporated to dryness. The 3,5-dinitrobenzyl alcohol metabolite is eluted from the Florisil column with dichloromethane and evaporated to dryness. The parent and metabolite residues are each dissolved in hexane prior to separate analysis using nitrogen-specific GLC/FID and a column packed with 3% OV-101 on Supelcoport 80/100 mesh. The reported limit of detection is 0.05 ppm for each compound.

Recoveries were 83.7-104.8% from 12 samples of soybean forage fortified with pendimethalin at 0.05-1.0 ppm. Recoveries were 83.3-112.7% from 11 samples of soybean forage fortified with the 3,5-dinitrobenzyl alcohol metabolite at 0.05-1 ppm; recovery was 69.4% from one sample fortified at 0.1 ppm. Apparent residues of pendimethalin and its metabolite (three control samples for each compound) were <0.05 ppm.

This method is adequate for data collection pertaining to the residues of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite in or on soybean forage samples.

Soybean forage and hay

The method recoveries reported above for soybean forage do not support the available data (MRID 40185101) pertaining to residues of pendimethalin and its 3,5-dinitrobenzyl alcohol metabolite in or on soybean hay and straw. Additional validation data are still outstanding for soybean hay and straw.

ATTACHMENT 2

DYNAMAC
CORPORATION

Final Report

PENDAMETHALIN (CBRS No. 7517) Task 4: Registrant's Response to Product Chemistry Data Requirements

March 22, 1991

Contract No. 68-D8-0080

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

Submitted by:
Dynamac Corporation
The Dynamac Building
2275 Research Boulevard
Rockville, MD 20850-3268

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PENDIMETHALIN (AMERICAN CYANAMID; CBRS NO. 7517)

REGISTRANT'S RESPONSE TO PRODUCT CHEMISTRY DATA REQUIREMENTS

Task - 4

BACKGROUND

The Pendimethalin Registration Standard Update of 3/19/90 requires additional data for the American Cyanamid 90% technical (T; EPA Reg. No. 241-245) pertaining to certified limits and enforcement analytical methods. In response, American Cyanamid has submitted a single volume of product chemistry data (CBRS No. 7517; 1990; MRID 41725201). These data and our conclusions pertain only to the certified limits and enforcement analytical methods for the American Cyanamid 90% T.

62-2. Certification of Limits

The Pendimethalin Registration Standard Update of 3/19/90 requires that nominal concentrations be reported on EPA Form 8570-4 (Rev. 2-85). In response, American Cyanamid has submitted a CSF dated 12/5/90 (1990; MRID 41725201) for the 90% T (EPA Reg. No. 241-245). Data are presented in Confidential Appendix A and do not satisfy the requirements of 40 CFR §158.175 (Guideline Reference No. 62-2) regarding certified limits for the American Cyanamid 90% T (EPA Reg. No. 241-245) because a nominal concentration for the active ingredient was not included on the Confidential Statement of Formula (CSF); a complete CSF, submitted on EPA Form 8570-4 (Rev. 2-85), is required to fulfill the outstanding data requirements. Additional data are required.

62-3. Enforcement Analytical Methods

The Pendimethalin Registration Standard Update of 3/19/90 requires additional validation for the analytical methods used to determine impurities of toxicological significance. In response, American Cyanamid has submitted (1990; MRID 41725201) additional validation data which are presented in Confidential Appendix B. These data satisfy the requirements of 40 CFR §158.180 (Guideline Reference No. 62-3) regarding enforcement analytical methods for the American Cyanamid 90% T (EPA Reg. No. 241-245). No additional data are required.

TABLE A. GENERIC DATA REQUIREMENTS FOR THE PENDIMETHALIN (AMERICAN CYANAMID) TECHNICAL GRADE OF THE ACTIVE INGREDIENT.[1]

Data Requirement	Test Substance[2]	Guideline Status	Must additional data be submitted under FIFRA Sec. 3(c)(2)(B)? [Yes]	[No]	Reference (MRID No.)
40 CFR §158.155-190 Product Chemistry					
Product Composition					
61-2. Beginning Materials and Production Process	TGAI	R		X	
61-3. Formation of Impurities	TGAI	R		X	
Analysis and Certification of Product Ingredients					
62-1. Preliminary Analysis	TGAI	CR		X	
Physical and Chemical Characteristics[3]					
63-2. Color	TGAI	R		X	
63-3. Physical State	TGAI	R		X	
63-4. Odor	TGAI	R		X	
63-5. Melting Point	TGAI	R		X	
63-6. Boiling Point	TGAI	R		X	
63-7. Density, Bulk Density, or Specific Gravity	TGAI	R		X	
63-8. Solubility	TGAI or PAI	R		X	
63-9. Vapor pressure	TGAI or PAI	R		X	
63-10. Dissociation Constant	TGAI or PAI	R		X	
63-11. Octanol/Water Partition Coefficient	PAI	CR		X	
63-12. pH	TGAI	CR		X	
63-13. Stability	TGAI	R		X	
Other Requirements:					
64-1. Submittal of Samples	TGAI or PAI	CR		X	

TABLE A. (Continued).

1. Data requirements pertain to the American Cyanamid 90% T (EPA Reg. No. 241-245). Additional data requirements are listed in the following Table B, "Generic Data Requirements for Pendimethalin Manufacturing-Use Products", for registered technical products.

2. Test substance: MP = manufacturing-use product; PAI = purified active ingredient; TEP = typical end-use product; TGAI = technical grade of the active ingredient.

3. As required by 40 CFR §158.190 and more fully described in the Pesticide Assessment Guidelines, Subdivision D, Guidelines Reference Nos. 63-2 through 63-13, data must be submitted on physicochemical characteristics (color, physical state, odor, melting point, boiling point, specific gravity, solubility, vapor pressure, dissociation constant, octanol/water partition coefficient, pH, and stability). There are additional data requirements listed in Table B pertaining to physicochemical characteristics of those technical products which are also manufacturing use products.

TABLE B. PRODUCT SPECIFIC DATA REQUIREMENTS FOR PENDIMETHALIN (AMERICAN CYANAMID) MANUFACTURING-USE PRODUCTS.[1]

Data Requirement	Test Substance[2]	Guideline Status	Must additional data be submitted under FIFRA Sec. 3(c)(2)(B)? [Yes]	[No]	Reference (MRID No.)
40 CFR §158.155-190 Product Chemistry					
Product Composition					
61-1. Product Composition	MP	R		X	
61-2. Beginning Materials & Production/Formulation Process	MP	R		X	
61-3. Formation of Impurities	MP	R		X	
Analysis and Certification of Product Ingredients					
62-1. Preliminary Analysis	MP	CR		X	
62-2. Certified Limits	MP	R	X[3]		41725201
62-3. Enforcement Analytical Methods	MP	R		X	41725201
Physical and Chemical Characteristics[4]					
63-2. Color	MP	R		X	
63-3. Physical State	MP	R		X	
63-4. Odor	MP	R		X	
63-7. Density, Bulk Density, or Specific Gravity	MP	R		X	
63-12. pH	MP	CR		X	
62-14. Oxidizing/Reducing Action	MP	CR		X	
62-15. Flammability	MP	CR		X	
63-16. Explodability	MP	R		X	
63-17. Storage Stability	MP	R		X	
63-18. Viscosity	MP	CR		X	
63-19. Miscibility	MP	CR		X	

(Continued, footnotes follow)

TABLE B. (Continued).

Data Requirement	Test Substance	Guideline Status	Must additional data be submitted under FIFRA Sec. 3(c)(2)(B)? [Yes]	[No]	Reference (MRID No.)
63-20. Corrosion Characteristics	MP	R		X	
Other Requirements:					
64-1. Submittal of Samples	MP	CR		X	

1. Data requirements pertain to the American Cyanamid 90% T (EPA Reg. No. 241-245). Additional data requirements are listed in the preceding Table A, "Generic Data Requirements for the Pendimethalin Technical Grade of the Active Ingredient", for the TGAI of manufacturing-use products.

2. Test substance: MP = manufacturing-use product; PAI = purified active ingredient; TEP = typical end-use product; TGAI = technical grade of the active ingredient.

3. American Cyanamid has responded to data requirements for the 90% T; however the nominal concentration of the active ingredient must be included on the Confidential Statement of Formula. Certifications must be submitted on EPA Form 8570-4 (Rev. 2-85).

4. As required in 40 CFR §158.190 and more fully described in the Pesticide Assessment Guidelines, Subdivision D, Guidelines Reference Nos. 63-2 through 63-20, data must be submitted on physicochemical characteristics of each manufacturing-use product (color, physical state, odor, specific gravity, pH, oxidizing or reducing action, flammability, explodability, storage stability, viscosity, miscibility, and corrosion characteristics). Additional data requirements regarding physicochemical properties of manufacturing-use products which contain only the technical grade of the active ingredient are listed in Table A, "Generic Data Requirements for the Pendimethalin Technical Grade of the Active Ingredient."

ATTACHMENT 3

PENDIMETHALIN (AMERICAN CYANAMID; CBRS NO. 7517)

PRODUCT CHEMISTRY

TASK 4

(Final Report)

CONFIDENTIAL APPENDICES

Appendix A: 2 Page(s)
Appendix B: 1 Page(s)

Confidential Appendices to the Scientific Review of a Registration Standard Followup Report for the pesticide pendimethalin by the Chemistry Branch II Reregistration Section [Confidential FIFRA Trade Secret/CBI].

Page _____ is not included in this copy.

Pages 19 through 21 are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product impurities.

✓ Description of the product manufacturing process.

✓ Description of quality control procedures.

_____ Identity of the source of product ingredients.

_____ Sales or other commercial/financial information.

_____ A draft product label.

_____ The product confidential statement of formula.

_____ Information about a pending registration action.

_____ FIFRA registration data.

_____ The document is a duplicate of page(s) _____.

_____ The document is not responsive to the request.

The information not included is generally considered confi by product registrants. If you have any questions, please the individual who prepared the response to your request.