

US EPA ARCHIVE DOCUMENT

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DATE OUT: NOV/10/1998

PRODUCT CHEMISTRY REVIEW OF A TECHNICAL GRADE OF ACTIVE INGREDIENT

DP BARCODE No.: 249731 EPA RECEIVED DATE: 29/MAY/1998 REG./File Symbol No.: 33660-

UN PRODUCT NAME: Pendimethalin: N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine

MRIDs: 445421-01C, 445421-01 to -09, 445560-02, 445570-01C to -09C & 445570-03 to -09

COMPANY NAME: Industria Prodotti Chimici S. P. A. Action Code: 166

FROM: Sami Malak, Chemist /S/ *Sami Malak*
Technical Review Branch/RD (7505C)

TO: 25 Jim Tompkins/Lisa Jones
Herbicide Branch/RD (7505C)

INTRODUCTION:

On behalf of Industria Prodotti Chimici S. P. A., Lewis & Harrison requests a "Me-Too" registration of this technical grade of active ingredient, claiming its similarity to [REDACTED]. In support of this request, the applicant included product chemistry data, product's CSF a basic formulation dated 15/MAR/1998, and label EPA received 29/MAY/1998. A Certification With Respect to Citation of Data was also included with this submission.

FINDINGS:

1. This technical grade of active ingredient, Pendimethalin: N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine, Reg. No. 33660-UN, CAS #40487-42-1, is similar to [REDACTED]. It is [REDACTED] purer than the registered product, 92.6% as opposed to [REDACTED].
2. The submitted product chemistry data is adequate and satisfy the requirements for a "Me-Too" registration of this technical grade of active ingredient.
3. An adequate GC analytical method is available for enforcement. The method entitled: Pendimethalin Technical And Formulated Products, GLC Determination of Pendimethalin In Technical And Its Formulations, Validation of The Analytical Method." The method, included in MRID #445570-01C, was authored by Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 12/SEP/1995, 107 pages. The

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method utilizes diisopropyl phthalate as an internal standard & is applicable to the determination of pendimethalin per se in pendimethalin technical grade of active ingredient and formulated products. Detailed considerations of the method, method accuracy, precision, sample calculation chromatograms, and validation data are to be found in this memorandum.

4. The ingredient statement, the physical/chemical hazard statement, and the storage and disposal statement cited on product's label, EPA received 29/MAY/1998, satisfy the requirements of 40CFR§156.10 & the nominal concentration as per the regulations of PR Notice 91-2.
5. The submitted product's CSF a basic formulations dated 15/MAR/1998 was filled out correctly and completely in compliance with the regulations of 40CFR§152.43. The nominal concentration of the active ingredient agrees with the label claim nominal concentration as per the regulations of PR Notice 91-2. Further, the upper and lower certified limits are within the standard limits of 40CFR§158.175(b)(2).

RECOMMENDATIONS

The applicant satisfied product chemistry data requirements for registration of this technical grade of active ingredient.

DETAILED CONSIDERATIONS

PRODUCT CHEMISTRY DATA REVIEW

1. A statement of data confidentiality dated 09/MAR/1998 was included with this submission claiming confidentiality of some of the data requirements on the basis of its falling within the scope of FIFRA§10(d)(1)(A), (B), or (C). Review of CBI information is to be found in Confidential Appendix A.
2. A GLP statement dated 09/MAR/1998 was included with this submission to the effect that some of the submitted studies were conducted in full compliance with GLP requirements of 40CFR§160.

DATA SUBMITTED

MRID #445441-01 The submitted study entitled "Product Identity and Composition of Pendimethalin Technical", was authored by Cristina Marchiori, Performed by I.Pi.Ci. Industria Prodotti Chimici S.p.A. of Novate Milanese, Italy, Completed 04/FEB/1998, 128 pages.

MRID #445570-01C The submitted study entitled "Pendimethalin Technical, Analysis and Certification of Product Ingredients", was authored by Cristina Marchiori, Performed by I.Pi.Ci. Industria Prodotti Chimici S.p.A. of Novate Milanese, Italy, Completed 05/FEB/1998, 125 pages.

MRID #445570-02 The submitted study entitled "Pendimethalin Technical And Formulated Products, GLC Determination of Pendimethalin In Technical And Its Formulations, Validation of The Analytical Method", authored by Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 12/SEP/1995, 107 pages.

MRID #445570-03C The submitted study entitled "Pendimethalin Technical,

Determination of Six Impurities In Technical Product By GC, Validation of The Analytical Method", authored by Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 04/MAR/1997, 149 pages.

MRID #445570-04C The submitted study entitled "Pendimethalin Technical, And Formulated Products Determination of N-NO-Pendimethalin Impurity Content By HPLC, Validation of The Analytical Method", authored by Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 27/FEB/1996, 71 pages.

MRID #445570-05C The submitted study entitled "Pendimethalin Technical, Determination of The Volatile Nitrosamines, Validation of The Analytical Method", authored by Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 23/JAN/1998, 71 pages.

MRID #445570-06C The submitted study entitled "Pendimethalin Technical, Quantitative Analysis Of The Impurities Not Detected By GC Analysis, authored by Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 19/JAN/1998, 34 pages.

MRID #445570-07C The submitted study entitled "Pendimethalin Technical, Silica Gel Column Separation Of Impurities Not Detected By GC Analysis And Their Identification, authored by Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 04/FEB/1998, 45 pages.

MRID #445570-08C The submitted study entitled "Pendimethalin Technical, Chemical Stability: Accelerated Storage Stability Test, author/study director: Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 04/FEB/1998, 277 pages.

MRID #445570-09C The submitted study entitled "Pendimethalin Technical, One Year Storage Stability Test Under Warehouse Conditions And Corrosion Characteristics, authored by Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 04/FEB/1998, 249 pages.

MRID #445421-02 The submitted study entitled "Pendimethalin Technical, Determination of Appearance Colour And Odour, Data Requirement, Guidelines 63-2, 63-3, 63-4", authored by Dr. R. Fabbrini, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 28/APR/1995, 15 pages.

MRID #445421-03 The submitted study entitled "Pendimethalin Technical, Determination Of The Melting Point, Data Requirement, Guidelines 63-5,

authored by Dr. R. Fabbrini, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 14/APR/1995, 20 pages.

MRID #445421-04 The submitted study entitled "Pendimethalin Technical, Determination Of The Relative Density, Data Requirement, Guidelines 63-7, authored by Dr. R. Fabbrini, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 28/APR/1995, 17 pages.

MRID #445421-05 The submitted study entitled "Pendimethalin Technical, Determination Of Water Solubility, Data Requirement, Guidelines 63-8, authored by Dr. R. Fabbrini, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 30/APR/1995, 33 pages.

MRID #445421-06 The submitted study entitled "Pendimethalin Technical, Determination Of The Solubility In Organic Solvents, Data Requirement, Guidelines 63-8, authored by Dr. R. Fabbrini, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 12/MAY/1995, 15 pages.

MRID #445421-07 The submitted study entitled "Pendimethalin Technical, Determination Of The Vapor Pressure, Data Requirement, Guidelines 63-9, authored by Dr. R. Fabbrini, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 04/MAY/1995, 39 pages.

MRID #445421-08 The submitted study entitled "Pendimethalin Technical, Determination Of Partition Coefficient, Data Requirement, Guidelines 63-11, authored by Dr. R. Fabbrini, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 08/MAY/1995, 36 pages.

MRID #445421-09 The submitted study entitled "Pendimethalin Technical, Determination Of Oxidizing Properties, Data Requirement, Guidelines 63-14, authored by Dr. R. Fabbrini, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 04/MAY/1995, 16 pages.

MRID #445421-10 The submitted study entitled "Pendimethalin Technical, Determination Of Absorption Spectra (UV, Vis, IRD, NMR, MS), Data Requirement, OPPTS 830-7050, authored by Dr. R. Fabbrini, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 28/APR/1995, 31 pages.

MRID #445573-01C The submitted study entitled "Analysis To Demonstrate The Similarity Between Pendimethalin Manufactured By I.Pi.Ci. And Pendimethalin Manufactured by American Cyanamid, authored by Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 04/DEB/1998, 9 + 175

pages.

Group A, Series 830-Product Identity, Composition, and Analysis (40 CFR 155, 160, 162, 167, 175 & 180)

830-1550 Product Identity and Composition

Chemical Name: N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine.

PPC No.: 108501

Common Name: Pendimethalin

EPA Reg. No.: 33660-UN

CAS Registry No.: 40487-42-1

Type: Herbicide

Uses: For formulation of Herbicides only.

Empirical Formula: $C_{13}H_{19}N_3O_4$

Structural Formula: Included in Confidential Appendix B.

Molecular Weight: 281.3

Supplier: Industria Prodotti Chimici S. P. A., Italy

Nominal Concentration..... 92.6

Upper Limit..... 95.0%

Lower Limit..... 90.0%

830-1600 Description of Materials Used to Produce the product

(See Confidential Appendix A).

830-1620 Description of Production Process

(See Confidential Appendix A).

830-1670 Discussion of Formation of Impurities

(See Confidential Appendix A).

Series 62 Analysis and Certification of Product Ingredients

830-1700 Preliminary Analysis

(See Confidential Appendix A).

830-1750 Certified Limits

(See Confidential Appendix A).

830-1800 Enforcement Analytical Method

Method for the Active Ingredient: MRID #445570-01C & 445570-02

An adequate GC analytical method is available for enforcement. The method entitled: "Pendimethalin Technical And Formulated Products, GLC Determination of Pendimethalin In Technical And Its Formulations, Validation of The Analytical Method." The method, included in MRID #445570-01C & 445570-02 was authored by Dr. S. Garofani, Performed by Chemservice S.P.A. of Novate Milanese, Italy, Completed 12/SEP/1995, 107 pages, Prepared and Validated by ChemService (Study CH-38/95)." The method utilizes diisopropyl phthalate as an internal standard & is applicable to the determination of pendimethalin per se in pendimethalin technical grade of active ingredient and formulated products.

In this method the internal standard is prepared by dissolving 80 mg each of pendimethalin technical and diisopropyl phthalate internal standard in acetone. Then 1 μ l of the resulting solution is injected into GC to determine the "F" factor.

The technical sample is prepared by dissolving 5 g of a sample in 15 ml in Solvesso. 300 μ l of this solution plus 80 mg of the internal standard are dissolved in 100 ml acetone. Then 1 μ l of the resulting solution is injected into GC to determine peak area.

Chromatographic Conditions:

GLC Column : HP1 5 m x 0.53 mm, film thickness 2.65 μ g
Detector : FID
Column temperature : 120°C
20°C/min from 120°C to 220°C
Injector temperature : 240°C
Detector temperature : 300°C
Column head pressure : 25 KPa at 120°C; split 160 ml/min ca. 8:1
Injection volume : 1 μ l
I.S. retention time : 2.1 min
AI retention time : 3.3 min

Method validation data, method accuracy and precision are adequate. Sample calculations and sample chromatograms were included with this submission.

Analysis of six batches gave an average of 93.28% for technical pendimethalin and 34.56% for the end-use product, Ipimethalin, Reg. No. 33660-GO.

Methods for Impurities: Please refer to Confidential Appendix A.

Group B, Series 830-Physical and Chemical Properties (40 CFR 158.190)

GUIDELINE REFERENCE NO.(GRN)/TITLE	VALUE OR QUALITATIVE DESCRIPTION/METHOD(S) USED WHERE APPLICABLE AND REFERENCES	MRID NO.
-6302 Color	Orange-brown.	445421-02
-6303 Physical State	Viscous liquid.	445421-02
-6304 Odor	Slightly aromatic.	445421-02
-6313 Stability to Normal and Elevated Temperature, Metals, and Metal Ions.	Stable to elevated temperature (54°C) for a period of 14 days.	445570-08
-6314 Oxidation/Reduction: Chemical Incompatibility	NA	
-6315 Flammability/Flame Extension	Not flammable.	CSF
-6316 Explodability	N/A.	
-6317 Storage Stability	Stable for a period of one year, the duration of testing.	445570-09C
-6319 Miscibility	N/A.	
-6320 Corrosion Characteristics	Non corrosive to the commercial container.	445570-09C
-6321 Dielectric Breakdown Voltage	N/A.	
-7000 pH	NA	
-7050 UN/Visible Absorption	Molar absorptivity of $4.9 \times 10^5 \text{ M} = 28775$ at 238 nm & 5490 at 425 nm.	445421-1003
-7100 Viscosity	NA	
-7200 Melting Point/Melting Range	53.4°C	445421-03
-7220 Boiling Point/Boiling Range	NA	
-7300 Density/Relative Density/Bulk Density	1.17772 g/ml	445421-04
-7370 Dissociation Constant in Water	NA	

-7520 Particle Size, Fiber Length, and Diameter Distribution	N/A.	
-7550 Partition Coefficient (n-Octanol/H ₂ O), Shake Flask Method	Log P _{ow} = 5.1	445421-08
-7560 Partition Coefficient (n-Octanol/H ₂ O), Generator Column Method	NA	
-7570 Partition Coefficient (n-Octanol/H ₂ O), Estimation by Liquid Chromatograph	NA	
-7840 Water Solubility: Column Elution Method; Shake Flask Method	0.021 g/100 ml	445421-05
-7860 Water Solubility: Generator Column Method	NA	
-7950 Vapor Pressure	1.94 to 7.99 mPa for PAI & 0.95 mPa for the TGA1	445421-07

MRID #445421-06: Solubilities in Organic Solvents in g/100 ml: n-hexane 4.898; n-Octanol 6.608; Dichloroethane >80; Xylene >80; Ethylacetate >80; and Acetone >80.

Attachments:

1. Confidential Appendix A: Pendimethalin Composition and Analysis (pages 9-14).
2. Confidential Appendix B: Description of Production Process and Names & structural of GC-Detectable Pendimethalin Impurities (pages 15-19).

cc: Sami Malak and Central File (Reg. No. 33660-UN).
7505C:RD:TRB:S.Malak:CM-2:Rm256:s.m.:11/NOV/1998:703-308-9365:<33660-UN>.

Page _____ is not included in this copy.

Pages 10 through 17 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.

X 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.

_____ Proprietary information pertaining to the chemical composition of an inert ingredient provided by the source of the ingredient.

_____ Attorney-Client Privilege.

_____ Claimed Confidential by submitter upon submission to the Agency.

_____ Internal Deliberative Information.

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