

US EPA ARCHIVE DOCUMENT

**DP BARCODE:** D329849; **Reg. No. /FILE SYMBOL No.:** 70506-REE; **PRODUCT:** UPI Imidacloprid Technical Insecticide (98%)

October 30, 2006

FEE

**SUBJECT:** FEE. Secondary Product Chemistry Review on UPI Imidacloprid Technical Insecticide

**FROM:** Shyam B. Mathur, Ph.D  
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*10/31/06*

**TO:** Daniel Dani / John Hebert, RM 07  
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*JH*

**DP BARCODE:** D329849  
**EPA REG. NO.:** 70506-REE  
**PRODUCT:** UPI Imidacloprid Technical Insecticide  
**PCC:** 129099  
**REGISTRANT:** United Phosphorous Incorporation (UPI)  
**USE:** Insecticide-Rodenticide

**INTRODUCTION:**

The registrant UPI has submitted an application for the registration of UPI Imidacloprid Technical produced at Ankleshwar, Gujarat, India. The registrant has submitted CSF for basic formulation (dated 05-09-06) and supporting product chemistry data under MRID Nos. 468335-01 and 468335-02. The registrant has claimed that the proposed UPI imidacloprid TGA/MUP is substantially similar to the registered product with Reg. No. 42750-104. The Dynamac Corporation conducted the primary review of the product chemistry data submitted (excluding CSF). TRB has been asked to perform the secondary review.

**SUMMARY OF FINDINGS:**

1. The CSF for basic formulation (dated 05-09-06) is filled out completely and correctly. The nominal concentration (NC) of the active ingredient (98%) concurs with the product label claim nominal concentration. The CSF is in compliance with PR Notice 91-2. The proposed certified limits for the AI are in compliance with standard certified limit table set-forth in 40CFR §158.175(b)(2). The proposed upper certified limits of the impurities are based on preliminary analysis and the potential for variations in the manufacturing process. The product chemistry data submitted corresponding to guideline reference 830.1550 (product identity & composition) and 830.1750 (certified limits) satisfy the data requirements of 40CFR§158.155 and 158.175 respectively [MRID No. 468335-01].
2. The product chemistry data submitted corresponding to guideline 830.1600 (description of materials use to produce the product) satisfy the data requirements of 40CFR§158.160. The registrant has provided the MSDS for all the starting materials used to produce the active ingredient [MRID No. 468335-01].
3. The product chemistry data submitted corresponding to guideline reference 830.1620 (description of production process) do not satisfy the data requirements for 40CFR§158.162. The active ingredient was produced in [REDACTED]. The production process has been described with detail, however some of the important information regarding the amounts of the chemical used in the each step and the yields of the product obtained in each step have not been provided [MRID No. 468335-01].

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4. The product chemistry data submitted corresponding to guideline reference 830.1670 (Discussion on the formation of impurities) satisfy the data requirements for 40CFR§158.167. The registrant has provided the mechanisms of formation and identification of all impurities identified in the 5-batch analysis at  $\geq 0.1\%$ . The registrant has listed [REDACTED] impurities on the CSF [MRID No. 468335-01].

5. The data submitted corresponding the guideline reference 830.1700 (Preliminary analysis) satisfy the data requirements of 40CFR§158.170. Five representative lots of the manufacturing concentrate were analyzed for weight percent of AI. Analyses were performed using HPLC-UV(252 nm) with internal standard method. Five representative batches of the 98% T were analyzed for percent AI and the impurities. Samples were analyzed [REDACTED] by HPLC/UV. Acceptable method validation data were submitted. The five-batch analysis supports the CSF [MRID No. 468335-01].

6. The data submitted corresponding the guideline reference 830.1800 (Enforcement Analytical method) satisfy the data requirements of 40CFR§158.180. The HPLC-UV (252 nm) with internal standard method was used for the determination of the AI [MRID No. 468335-01].

7. The product chemistry data submitted corresponding to guideline reference 830 Series Subgroup B (physical/chemical properties) for the UPI imidacloprid technical insecticide satisfy the data requirements of 40CFR§158.190, except for the guidelines 830.6317 (storage stability), and 830.6320 (corrosion characteristics).

#### CONCLUSIONS

TRB has reviewed the product chemistry data submitted for 830 series Subgroup A & Subgroup B for UPI Imidacloprid technical and has concluded that:

1. The CSF for basic formulation (both 05-09-06) is acceptable.
2. The product chemistry data submitted corresponding to guidelines 830 series subgroup A are acceptable, except for the guideline 830.1620 (description of production process). The registrant must provide information regarding the quantities of starting chemical used in each step of the synthesis along with the production yield in each step.
3. The product chemistry data submitted corresponding to guidelines 830 series subgroup B are acceptable, except for the guidelines 830.6317 (storage stability), and 830.6320 (corrosion characteristics).
4. The registrant must submit the results of one year storage stability (830.6317) and corrosion characteristics (830.6320) to the Agency on completion. It is recommended that the observations must be made at 0, 3, 6, 9, & 12 month intervals. The results must be submitted in a hard copy and an electronic format is also requested.
5. The proposed technical (File Symbol No. 70506-REE) was determined to be substantially similar to the registered product with Reg. No. 42750-104 from product chemistry point of view.

**Note to RM:** The reviewer tried two times to contact the registrant regarding the issue with the guideline 830.1620 but no response was received.

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830.1550. Product identity & Composition: (MRID No. 46833501)

Common Name: Imidacloprid

Chemical name (CAS): 1-[(6-chloro-3-pyridinyl)methyl]-N-nitro-2-imidazolidinimine  
(IUPAC): (EZ)-1-(6-chloro-3-pyridylmethyl)-N-nitroimidazolidin-2-ylideneamine

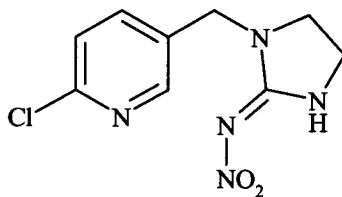
CAS No.: 138261-41-3

PC Code No.: 129099

Empirical formula: C<sub>9</sub>H<sub>10</sub>N<sub>5</sub>ClO<sub>2</sub>

Molecular Weight: 255.7

Structural formula



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 Imidacloprid Technical Insecticide (98%)

Table 1. Manufacturing and Impurity Data for the United Phosphorous, Inc. 98% TGAI/MUP				
GLN	Requirement	MRID	Status	Details and /or Deficiency
830.1550	Product Identity and composition	46833501	A	The NC of the AI (98%) is supported by the 5-batch analysis and agrees with the label claim. Impurities >0.1% are listed on the CSF.
830.1600	Description of materials used to produce the product	46833501	A	The identity of the starting materials used to produce the technical product has been provided by the registrant.
830.1620	Description of production process	46833501	U	The 98% T is produced in [REDACTED] of the AI. Additional information is required concerning the quantity of product produced per theoretical batch size, and relative quantities in pounds or kilograms for each ingredient used in the process.
830.1670	Discussion of formation of impurities	46833501	A	The registrant has provided the mechanisms of formation and identification of all impurities identified in the 5-batch analysis at $\geq 0.1\%$ , as well as [REDACTED]
830.1700	Preliminary analysis	46833501	A	Five representative batches of the 98% T were analyzed for percent AI and the impurities. Samples were analyzed for [REDACTED] by HPLC/UV. Acceptable method validation data were submitted. The five-batch analysis supports the CSF.
830.1750	Certified limits	46833501	A	The proposed certified limits for the AI are based on the standard certified limits of $\pm 3\%$ of the nominal concentration. The proposed upper certified limits for the impurities were based on preliminary analysis results and the potential for variations in the manufacturing process.
830.1800	Enforcement analytical method	46833501	A	The methods submitted for preliminary analysis are proposed for enforcement purposes. The AI is determined by a published HPLC/UV method, CIPAC Method 582. Impurities are determined by HPLC/UV and ion chromatography methods.
A = Acceptable; N = unacceptable (see Deficiency); N/A = Not Applicable; G = Data gap; I = In progress or need upgrade; U = Up-grade (additional information required)				

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830 Series Subgroup B (Physical-Chemical Properties)

Table 2: Physical and Chemical Properties of: United Phosphorous, Inc. 98% TGAI/MUP				
GLN	Requirement	MRID	Status	Result or Deficiency [Test substance; method]
830.6302	Color	46833502	A	White [TGAI/MUP]
830.6303	Physical state	46833502	A	Powder [TGAI/MUP]
830.6304	Odor	46833502	A	Odorless [TGAI/MUP]
830.6313	Stability to normal and elevated temperatures, metals, and metal ions	46833502	A	No change in appearance (color, odor, physical state, and pH) or the percent active ingredient after storage at 54 °C for 14 days. [TGAI; CIPAC MT 46.1.1.1]  Waiver requested for stability to metals and metal ions because the technical product will be packaged, stored, and shipped in fiber board drums lined with HDPE; therefore, there is no contact with metals or metal ions. The Agency recommends for this waiver.  Data are required demonstrating stability of the TGAI at ambient temperature.
830.6314	Oxidation/reduction: chemical incompatibility	46833502	N	"Not applicable for the TGAI" Data are required for the T/MUP.
830.6315	Flammability	46833502	N	"Not applicable for the TGAI" Data are required for the T/MUP.
830.6316	Explosibility	46833502	N	"Not applicable for the TGAI" Data are required for the T/MUP.
830.6317	Storage stability	46833502	I	A one-year study is in progress; the final report will be submitted upon completion.
830.6319	Miscibility		N/A	The T/MUP is a solid at room temperature
830.6320	Corrosion characteristics	46833502	I	A one-year study is in progress; the final report will be submitted upon completion.
830.7000	pH	46833502	A	7.42 at 20 °C (1% w/w) [TGAI/MUP; CIPAC MT 75.3]
830.7050	UV/Visible absorption	46833502	A	See Note 1
830.7100	Viscosity		N/A	The T/MUP is a solid at room temperature.
830.7200	Melting point	46833502	A	143.6 °C [99.04% PAI; OECD No. 102]
830.7220	Boiling point		N/A	The TGAI is a solid at room temperature.
830.7300	Density	46833502	A	Specific gravity = 1.54 g/cm <sup>3</sup> at 20 °C [TGAI; CIPAC MT 3.2.1]
830.7370	Dissociation constants in water (DC)	46833502	A	Does not dissociate, and no dissociation constant could be determined. [99.04% PAI; pH changes on titration with 0.01 M HCl]
830.7550	Octanol/water partition coefficient	46833502	A	Log K <sub>ow</sub> = 0.57 [99.04% PAI; A.I. determination by HPLC/UV]
830.7840	Water solubility	46833502	A	At 20 °C: 0.61 at pH 5 0.59 at pH 7.0 0.58 at pH 9.0 [99.04% PAI; A.I. determination by HPLC/UV]
830.xxxx	Solvent solubility	46833502	A	At 20 °C: 0.07 g/L n-heptane 0.10 g/L p-xylene 3.08 g/L ethyl acetate 5.20 g/L methanol 10-14 g/L 1,2-dichloroethane 25-29 g/L acetone [99.04% PAI; CIPAC MT 181 and 157]
830.7950	Vapor pressure	46833502	N	"Cite-all"; 4 x 10 <sup>-7</sup> mPa at 20 °C (literature value) Data are required for the imidacloprid PAI produced by United Phosphorous, Inc.

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GLN	Requirement	MRID	Status	Result or Deficiency [Test substance; method]
A = Acceptable; N = unacceptable (see Deficiency); N/A = Not Applicable; G = Data gap; I = In progress or need upgrade; U = Up-grade (additional information required); W = waivers				

Note 1. 830.7050 UV/visible absorption  
For the 98.08% TGAI at ambient temperature [spectrophotometer]:

Nature of solution; pH	Absorption maxima, wavelength (nm)	Absorbance	Molar Extinction Coefficient, $\epsilon$
Neutral; 6.75	212.0	0.677	$1.34 \times 10^4$
	269.0	1.096	$2.17 \times 10^4$
Acidic; 1.64	212.0	0.635	$1.31 \times 10^4$
	269.0	1.034	$2.13 \times 10^4$
Basic; 12.46	218.0	0.609	$1.26 \times 10^4$
	269.0	0.708	$1.46 \times 10^4$

830.1800. Enforcement Analytical Method: (MRID 46833501)

HPLC/UV CIPAC Method No. 582/TC/(M) is proposed as the enforcement analytical method for the active ingredient in the imidacloprid technical. Imidacloprid was quantitated in the preliminary analysis study using a similar method (without an internal standard).

Analytical CIPAC Method 582 for determination of the active ingredient, imidacloprid

HPLC operating conditions:

Instrument: HPLC able to generate more than 7 Mpa pressure

Detector: UV (252 nm)

Column: 250 x 4.6 mm (i.d.) packed with 5  $\mu$ m octadecylsilane bonded silica gel

Column temperature: Ambient

Mobile phase: Isocratic, acetonitrile:water (60:40, v:v)

Flow rate: 1.2 mL/min

Sample size: 1  $\mu$ L

Calibration: External standards of imidacloprid with the internal standard propiophenone; peak area

Retention time: ~2.1 minutes imidacloprid; ~4.1 minutes internal standard (propiophenone)

Sample preparation: Technical samples are prepared with internal standard and acetonitrile.

No validation data were provided for the CIPAC method; however, because this is an established (published) method for imidacloprid, no validation data are required for the United Phosphorus technical.

Attachment: Confidential Appendix



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Pages 7 through 13 are not included in this copy.

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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.
  - 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.
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\* 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.

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