

US EPA ARCHIVE DOCUMENT

Date: October 8, 2003

SUBJECT: Product Chemistry Review of Mepiquat Chloride Technical

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S. Mathur
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DP BARCODE: D290649
EPA REG. NO.: 75095-R Mepiquat Chloride Technical
PCC : 109101
REGISTRANT: Hide, LLC
USE: Fungicide (Plant Growth Regulator)

INTRODUCTION:

The registrant Hide LLC, has submitted product chemistry in support of the registration application for the product Mepiquat chloride technical. The registrant has submitted a CSF for basic formulation (dated 4/14/03) and the product label. The product chemistry data have been submitted under MRID Nos. 459170-01, 459170-02, and 459378-01. The applicant has claimed that the proposed product is substantially similar to the registered product with Reg. No. 51036-187. The TRB has been asked to evaluate the product chemistry data submitted and determine whether data satisfy the requirements for registration with the Agency.

SUMMARY OF FINDINGS:

1. The registrant has submitted the Confidential Statement of Formula for basic formulation (dated 04-14-03), for Mepiquat chloride technical. The nominal concentration (99.0%) of the AI concurs with the product label claim nominal concentration of 99.0%. 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.
2. The product chemistry data submitted corresponding to guideline reference 830.1600 (Description of material used to produce the product) satisfy the data requirements of 40CFR§ 158.160. The registrant has provided the product specifications data on all the starting materials used for the production of this plant growth regulator. [MRID No. 459170-01]
3. The product chemistry data submitted corresponding to guideline reference 830.1620 (Description of production process) satisfy the data requirements for 40CFR§158.162. The details of the manufacturing process have been provided for the technical produced by [REDACTED] [MRID No. 459170-01]
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 described the formation of [REDACTED] main impurity present in the technical and other minor impurities. [MRID No. 459170-01]
5. The data submitted corresponding the guideline reference 830.1700 (Preliminary analysis) satisfy the data requirements of 40CFR§158.170. Five representative batches of the technical were analyzed for percent active ingredient using and the organic impurities by HPLC/MS (Selective Positive Ion Monitoring at m/z 114) method. The method was validated for accuracy, linearity, and precision. [MRID No. 459170-02]
6. The data submitted corresponding the guideline reference 830.1800 (Enforcement Analytical method) satisfy the data requirements of 40CFR§158.180. The HPLC / MS (PIM at m/z 114) method was used to assay the active ingredient in the technical. The method was validated for accuracy, linearity, and precision. [MRID No. 459170-01]

CONFIDENTIAL - ALL SOURCE INFORMATION IS NOT INCLUDED

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7. The registrant has submitted product chemistry data corresponding to guideline reference 830 Series Subgroup B (Physical/Chemical properties) for the technical produced in China. The data submitted satisfy the data requirements of 40CFR 158.190. [MRID No. 459378-01]

CONCLUSIONS:

The TRB has reviewed the product chemistry data submitted for the Mepiquat chloride technical and has concluded that:

1. All the product chemistry data submitted corresponding to 830 Series Subgroup A and Subgroup B satisfy the data requirements of 40CFR§158.155 to 158.190 and are acceptable, except for one year storage stability (830.6317) and corrosion characteristics (830.6320).
2. The CSF for basic formulation (dated 04-14-03) is filled out correctly or completely. The nominal concentration of the active ingredient (99.0%) concurs with the product label claim nominal concentration 99.0%. The CSF for basic formulation is acceptable.
3. Melting Point (830.7200) : The registrant has reported melting point for the test substance as 341.4°C, whereas in the literature the m.p. for mepiquat chloride reported is 223°C. This discrepancy must be addressed.
4. The registrant is advised to conduct one storage stability (830.6317) study on the proposed product under warehouse conditions with test substance stored in commercial container. The corrosion characteristics (830.6320) study may be conducted simultaneously. The observations and the assay for the AI must be conducted at intervals of 0, 3 months, 6 months, 9 months and 12 months.
5. The proposed product was determined to be substantially similar to the registered product with Reg. No. 51036-187 from product chemistry view point only.

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830.1550. Product Identity: [MRID No.: 459170--01]

Common Name: Mepiquat chloride

Chemical Name: 1,1-dimethylpiperidinium chloride

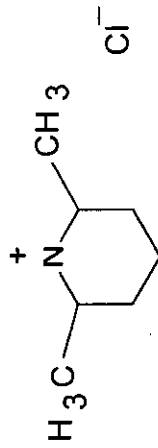
CAS No.: 24307-26-4

PC Code No.: 109101

Empirical formula: C₇ H₁₆ Cl N

Molecular Weight: 149.7

Structural formula:



Mepiquat Chloride

ATTACHMENT II

ATTACHMENT II

REVIEW OF PRODUCT CHEMISTRY, OPPTS 830 SERIES

Chemical Name (IUPAC, CAS)	Mepiquat Chloride technical
Chemical Number (CAS; PC Code)	CAS No. 24307-26-4 PC Code: 109101
Registration/Symbol No.	75095-R
Type of Product (T, MP, EP)	99.0% TGAI/ MP
DP Barcode	D290649
Reviewer	Shyam B. Mathur
Branch Chief	Deborah McCall

GLN	Requirement	MRID	Status ¹	Details and/or Deficiency ²
830.1550	Product identity and composition	Basic CSF (04-14-03)	A	The NC of AI (99.0%) is supported by 5 batch analysis & concurs with the product label claim 99.0%.
830.1600	Description of materials used to produce product	459170-01	A	The product specification sheets(MSDS) for all the starting materials have been provided by registrant.
830.1620	Description of production process	459170-01	A	The production process has been described in full details. The reaction conditions, amounts of the reagents and the equipment used in each step have been provided.
830.1670	Discussion of formation of impurities	459170-01	A	The registrant has provided the complete mechanisms of formation, quantification and identification of all the impurities. Xylene a toxicologically significant impurity has been identified and quantified and is present at levels of < 0.1%.
830.1700	Preliminary analysis	459170-02	A	Registrant has provided 5 batch analysis for the TGAI. The AI was assayed by using HPLC / MS (PIM at m/z 114). The impurities were also identified by using HPLC/MS methods. The analytical methods have been validated for precision, linearity & accuracy.
830.1750	Certified limits	Basic CSF (04-14-03)	A	The proposed certified limits for the TGAI were within standard certified limits. The NC & UCL for the tox concern impurities have been provided.
830.1800	Enforcement analytical method	459170-01	A	The HPLC / MS (PIM at m/z 114) method was used for the assay of the AI in technical.

A = Acceptable; N = Unacceptable (see Deficiency); N/A = Not Applicable.; G = Data gap; I = In progress or need upgrade;

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Table 2: Physical and Chemical Properties of Mepiquat Chloride Technical. *See Note for acceptance condition				
GLN	Requirement	MRID	Status ¹	Result ² or Deficiency
830.6302	Color	459378-01	A	Off white
830.6303	Physical state	" "	"	Crystalline powder
830.6304	Odor	" "	"	Characteristic aromatic odor
830.6313	Stability to normal and elevated temperatures, metals, and metal ions	cited	A	See Note 1 (*)
830.6314	Oxidation/reduction: chemical incompatibility	cited	A	None (*)
830.6315	Flammability		NA	
830.6316	Explosibility		NA	
830.6317	Storage stability			No data provided
830.6319	Miscibility		NA	
830.6320	Corrosion characteristics			No data provided
830.7000	pH	459378-01	A	6.220 (1% aqueous solution) @ 20 °C
830.7050	UV/Visible absorption	" "	A	No resolvable peaks observed
830.7100	Viscosity	NA		
830.7200	Melting point/ Melting range	459378-01	U	341.4 °C. The literature reported MP of 223 °C. There is a significant difference in values.
830.7220	Boiling point/ Boiling range		NA	
830.7300	Bulk Density	459378-01	A	Pour density=0.49 g/ml @ 20 °C Tap density=0.63 g/ml @ 20 °C
830.7370	Dissociation constants in water	" " "	A	Could not be measured by any of the methods
830.7550	Partition coefficient (n-octanol/water), shake flask method	cited	A	Ko/w = <10 (*)
830.7840	Water solubility: column elution method; shake flask method	459378-01	A	water = 650 g/L; MeOH=378 g/L; Hexane=<0.1 g/L; n-octanol=<0.1 g/L
830.7950	Vapor pressure	cited	A	<2.3 x 10 ⁻⁵ Torr at 25.3 °C (*)

A = Acceptable; N = Unacceptable (see Deficiency); N/A = Not applicable ; G = Data gap ; W = waiver request
I = Incomplete or in progress; U = Needs upgrading
(*) These data have been taken from product with Reg. No. 51036-187 (PCR 06/09/920) MRID Nos. 422319-03 & -04

Note 1. 830.6313. The samples were stored at ambient temperature and humidity for a period of 2 weeks mixed with metal, metal salts, or exposed to simulated sunlight. Analysis for reactions or degradation showed no apparent changes.

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830.1800. Enforcement analytical method: (MRID No. 459170-01)

The HPLC/MS method was used to determine the mepiquat chloride in the technical. This is the same method which was used for 5 batch analysis.
Instrumentation

HPLC System: ThermoSeparations, P4000 Pump, AS3000 Autosampler and Vacuum degasser.

Mass Spectrometer Detector, Finnigan-Mat, LCQ with electrospray source and Navigator Software.

HPLC/MS Parameters

Mobile Phase: 80/10/10 : Water/ACN/Water (pH = 3)

Flow rate: 0.5 ml/minute

Column: Zorbax C-3

Column temperature: 30°C

Injection volume: 5 µL

MS source: Electrospray

Capillary temperature: 250°C

Sheath Gas: 100; Aux gas: 60

MS Mode: Selective Positive Ion Monitoring at m/z 114 [The MS should be tuned specifically for mepiquat chloride detection at m/z 114.

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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.

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.
